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MINISTRY OF MUNITIONS

AND

DEPARTMENT OF SCIENTIFIC AND
INDUSTRIAL RESEARCH

Technical Records of Explosives Supply

1915-1918

No. 7

MANUFACTURE OF NITRIC ACID FROM NITRE AND SULPHURIC ACID

The following reports dealing with other aspects of the work of the Department of Explosives Supply have already been published by His Majesty's Stationery Office:—

Copies of the above and of the present series of reports may be obtained through any bookseller or direct from H.M. Stationery Office at the addresses given on the back of the cover.

PREFATORY NOTE

This is the seventh of a special series of reports which are being published in order to make available, for the benefit of the industries concerned, results of scientific and industrial value contained in the technical records of the Department of Explosives Supply of the Ministry of Munitions (*see list below*). The work recorded in these Reports was done at, or in connexion with, some of the National Factories during the war. The preparation of the necessary abstracts of information was begun by the Ministry of Munitions at the close of the war, and arrangements were afterwards made by the Department of Scientific and Industrial Research to complete them. The Department wish it to be clearly understood that the interesting information contained in this series of reports is the result of the labours of the Ministry of Munitions, and has been compiled by Mr. W. Macnab, C.B.E., F.I.C., an officer of that Ministry attached to the Department for this work.

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January 1922.

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INTRODUCTION

The production of nitric acid from nitre and sulphuric acid was carried out on a very large scale during the war.

Two factories alone, Queen's Ferry and Gretna, were able to turn out about 1,300 tons of nitric acid per week.

Again, the scarcity of experienced staff and workpeople along with the incessant demand for output caused many difficulties in the early days. The conditions for working the plants to the best advantage, however, were carefully studied and investigated, and soon the staff and workers became more expert and were able to conduct the operations with greater care and knowledge until excellent results were steadily obtained at nearly all the factories.

A description is given of the plant at Gretna and of its operations when all was running smoothly, then follow reports of some special trials and investigations which were of the greatest help to all concerned.

The recovery of nitric acid from the spent acid from the manufacture of nitrocotton by distillation is also described.

Finally, the recovery of nitre from the bags by washing is treated. This is important on account of the value of the nitre recovered and also as a means of diminishing the fire risk from bags impregnated with nitre, since several serious fires occurred from sparks alighting on unwashed nitre bags.

SECTION 1

DESCRIPTION OF PLANT AND PROCESS FOR MANUFACTURE OF NITRIC ACID BY RETORT PROCESS*

General outline.—Sodium nitrate (nitre) and sulphuric acid are heated in retorts by means of producer gas. Nitric acid is distilled off, condensed and collected, leaving a residue of nitre cake, which is ultimately run out of the retorts and collected.

DESCRIPTION OF BUILDINGS AND PLANT

Nitre stores.—Three, stone-built sheds, each 212 feet × 5 feet wide × 24 feet high (to eaves), with concrete floors and corrugated iron roofs, are provided. Each shed has a working capacity of 5,000 tons. Nitre stocks are brought into the sheds by railway wagons on elevated standard rail gauge (4 feet 8½ inches) tracks (two to each shed) built on stone piers. The nitre is dumped out of its containing bags on to the floor of the shed. Running along the floor of each shed are three sets of 18 inch gauge bogie tracks to provide a means of trucking the nitre to the drying machine and to the retort houses. The floors of the sheds are drained to small sumps, whence liquors can be recovered and transferred to the bag-washing plant for evaporation.

Cummer drier.—Fig. 1 shows the general arrangement and setting details of the Cummer nitre drier, two of which are installed.

The drier consists of a cylindrical steel tube, 27 feet long × 54 inches diameter, set at an inclination of ¾ inch in 12 inches towards the discharge end. The lower or discharge end is fitted with a cast-iron trunnion running in a bearing whilst the upper or feed end is fitted with a cast-iron ring supported on two cast-iron rollers. A third roller bearing on the back of the ring serves to take the thrust due to the inclination of the tube.

The front end of the tube is fitted with a spur ring driven by gearing and belt from a countershaft carried on top of the setting. This driving mechanism serves to rotate the tube at a speed of six revolutions per minute.

The tube is set in a brickwork chamber as shown, the covering consisting of fire-bricks laid on transverse 2½ inch × 3 inch × ⁵⁄₆ inch tees. At the discharge end of the drier a chute is built into the brickwork through which the dry nitre is discharged. The drier is coal-fired, being fitted with an automatic stoker driven from the above-mentioned countershaft, the combustion chamber being built out in front of the drier. The gases resulting from the combustion of the fuel pass into the brick chamber in which the drying tube is housed, and thence enter the discharge end of the tube passing over the nitre in the tube and out at the upper end of the tube. Only a small proportion of the gas is

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* The following descriptions refer more particularly to the plant at Gretna.

circulated around the outside of the tube, a fire-brick arch with perforations in the crown being built under the tube for the greater part of its length, thus causing the major portion of the heat to be transferred directly to the nitre in the tube. The gas leaving the drier contains a proportion of nitre in the form of dust, and is therefore passed through a dust chamber, this consisting of a brick chamber about 12 feet $\times$ 4 feet $\times$ 17 feet high, divided into two by a central vertical division wall, the gas entering at the top of one division, passing down same and entering the other division by an opening in the bottom of the division wall and thence out of a 3 foot diameter stack erected on top of the chamber. The tube is put under a slight suction by means of a fan rotating in the gas pipe at its entrance to the dust chamber, the fan being driven by belt from the main countershaft.

The furnace is provided with an additional chimney, 18 inches in diameter, with a damper and steam jet at the bottom for starting up and regulating purposes.

Particular care must be taken in building the firebrick arch below the tube, the bricks being rubbed to a good bearing and laid dry.

The wet nitre is brought into the drier house from the nitre store in hopper bottomed buckets by means of an overhead runway and is discharged from the bucket into a feed hopper, the feeder portion of which is below floor level in order that the bucket can run straight in without being hoisted. The feeder discharges the nitre into the boot of a wooden-cased elevator which elevates the nitre into a chute leading into the drier itself.

The nitre then passes through the drier coming in direct contact , with the fire gases, and is delivered dry into the boot of a second vertical elevator made with a steel casing.

This elevator lifts the dried nitre up to a sufficient height to discharge into the centre of a circular storage bin.

The bin is 16 feet diameter inside, having a capacity of 70 tons of nitre, and is built of brick in cement reinforced by circumferential tension hoops. The bottom of the bin is built with a conical bottom to facilitate discharge.

The runway bringing nitre into the drier house is connected up to that taking it out of the house, so that the wet nitre may be taken direct to the retort house should the drier be laid off for repairs.

The floor of the drier house is of conciete.

Each drier is capable of drying 80 tons of nitre per 24 hours. reducing the moisture from 2·5 to 0·5 per cent.

The following figures are taken from reports :—

- Fuel consumption (coke) per ton nitre handled = 0·014 ton.
- Wet nitre, moisture - - - = 2·5 per cent.
- Dry nitre, moisture - - - . = 0·5 per cent.

There is a tendency for the drum or drying chamber to get caked with hard nitre. This is more pronounced in wet weather. The remedy is to run through, say, once daily, 20 or 30 iron balls—eight-faced and pointed. This treatment generally removes the bulk of the scale.

The bottom of the storage bin is fitted with six discharge chutes
1 foot square, which, when open, deliver the nitre into bogies. Each
bogie passes over a weigh-bridge, and the contents are adjusted until
10 cwt. dry nitre are in the wagon. This is then taken to the retort
house and raised by means of an electric lift to the retorts' charging
platform.

Two electrical lifts are provided, one at the end of each house.
Each is driven by a 12 h.p. motor, and lifts a net load of 70 cwt. To
avoid corrosion the electric controls are placed in a separate shed, out
of the way of acid fumes.

The bogies containing the nitre run on a track on the charging
platform. The nitre is tipped into a movable chute fitting into the
man-hole of the retort, into which it is raked.

Sulphuric acid storage and feed tanks.—Two 9 foot × 30 foot boiler
tanks, elevated on brick piers, are provided for the storage of sulphuric
acid. From these tanks the acid flows by gravity into the retort houses
and communicates with the sulphuric acid feed tanks, one of which is
attached to each retort.

These feed tanks are mild steel cylindrical tanks 53 inches deep
× 51 inches diameter, and conveniently hold 2 tons of sulphuric acid.
Each is provided with a lid, gauge glass, and inlet and outlet valves.
From experience it has been decided that the gauge glasses fittings
are too small and the gauge glasses are not reliable. All tanks are
dipped before and after use, 1 inch of acid being equal to 0·059 tons
92 per cent. sulphuric acid. The tank has a sloping bottom to the
outlet valve. Each tank is elevated and sits on brickwork standing
30 inches high. The whole sits on a leaden tray 6 feet × 6 feet ×
2 inches, with a 1 inch diameter exit hole which connects to a lead
launder 4 inches × 4 inches, and in turn connects to the drain. The
outlet from the sulphuric acid measuring tanks is by a 2 inch lead pipe,
which connects to a permanent feed pipe fitted up to the retort lid.
This feed pipe is of cast iron and is flanged to the lead delivery pipe from
the tank. Connection into the retort is made by a T-piece, which is
provided with a blank flange for cleaning purposes.

In order to do away with the errors occurring in dipping these tanks,
an improved method has been developed. The same tank was used.
A hole was cut in the side, about 4 feet 3 inches from the bottom of the
tank, and an overflow pipe was inserted. Next an iron plunger was
placed inside, connected with a pulley and chain to which was attached
a pointer-adjacent to a scale. When the tank was to be used it was
filled up to overflowing, and, according to the strength of acid and nitrate
content of the nitre, the plunger inside the tank was adjusted by means
of the screw. This gave a definite volume of acid for each charge of
nitrate. This style of tank does very well, and when in use can be
depended upon to give nitre cakes of even acidities.

Nitric acid retorts.—The retorts are contained in two houses each
holding 24 retorts. They are built in each house in sets of three, the
brickwork settings of these batteries being separated by passages
2 feet 9 inches wide.

Fig. 2 shows the detail of the retort setting.

A raft of concrete is laid down sufficiently long to take a bank of three retorts and of suitable thickness depending on the nature of the ground.

On top of this the setting is built up as indicated.

It will be seen from this drawing that the gas in its course from the inlet to the chimney culvert 'is' caused to sweep around the retort in three complete turns and thence over the top of the retort and from there into the downtake and thence to the chimney culvert. The downtake is 12 inches × 9 inches and the chimney culvert is a semi-arch flue 3 feet × 3 feet 6 inches high. This flue is built in ordinary brick laid in cement, and extends the full length of the building, a branch at its middle point being taken off to the chimney. Suitable man-hole frames are built into the crown of the flue at intervals of about 30 feet.

All brickwork in the retort setting proper in contact with the hot gases is of the best firebrick, whilst all other brickwork above the floor level is laid in lime mortar.

The drawing also gives details of the gas culvert, this being a semi-arch culvert 3 feet wide × 3 feet 6 inches high lined with 4½ inches of the best firebrick. From this culvert at each retort is taken off a valve connection to the furnace, this valve being operated from the floor level or from the top of the retort setting. The gas after passing the control valve passes through a rectangular conduit 5 inches × 6½ inches and thence through a rectangular opening 6 inches × 3 inches. On leaving this opening it is mixed with the air necessary for combustion and enters the combustion chamber through a rectangular opening 9 inches × 6 inches. The quantity of air admitted for combustion is controlled by means of a slide valve.

Sixteen soot doors 12 inches × 15 inches are built into the setting of each retort as shown for cleaning out the gas passages, these doors being so designed as to serve also as explosion doors.

Particular care must be taken in laying all the firebrick linings of these settings to leave a clear space of at least ¾ inch between the lining and the ordinary brickwork of the setting to accommodate expansion of the retort and firebrick due to increase in temperature of these parts. This is important, as if neglected it will be found that the brickwork setting will crack when the retorts are operated.

The concrete rafts upon which these retorts are erected must be kept separate and distinct to allow of settlement of any bank of retorts. If this is not attended to any settlement which may occur after erection would likewise cause the brickwork of the settings to be distorted and cracked.

These retorts are gas-fired from the side nearest the condensers and tapped from the opposite side, so that these passages are useful as providing a way of communication for tappers who need to examine the fires, or for firemen who need to adjust the dampers. Immediately behind the retorts, and about 1 foot above the brickwork top, is a steel plate charging platform carrying the bogie tracks from the lifts, from which the bogies of nitre are conveyed to the retorts. The retort is of

cast iron, and consists of a lower portion, cylindrical in shape, with a rounded bottom and a cylindrical extension ring 36 inches high, the two parts being made one by a rust joint. The lid is very slightly dome-shaped, and fits into a collar in the extension ring, connection being made by means of a rust joint. The lid has a large hole about 2 feet 8 inches diameter, to which is bolted, by means of 15 1 inch bolts, the iron casting which embodies the changing man-hole, which is closed by a man lid, and the 8 inch diameter circular hole into which is fitted the uptake to the condensing system. The total capacity of the retort is about 9½ tons of water. This size is found suitable for the decomposition of 2 tons of sodium nitrate.

At the lowest end of the retort is a 2 inch outlet for the nitre cake. The outlet extends by a pipe to about 1 inch outside the brickwork setting. Round the end of the tapping pipe is fitted a collar which carries a screw-plug holding device to keep in position a cast-iron plug used to close the hole, an acid-tight plug being first made of damp clay. A later type of retort has a different kind of tapping hole, being 4 inches in diameter at the end, and instead of having a block of iron as a plug, has a swing-door, which is kept in position by means of a screw. This is a decided improvement on the old pattern.

The retorts are gas-fired by producer gas, which is led from the main gas flue through a 4 inch pipe controlled by a valve to a horizontal passage, 8 inches square, beneath the retort. A large amount of tar condenses in the 8 inch passage, and is drawn off through a cleaning door. The gas then passes up through a square hole into a brickwork combustion chamber, which is approximately 22 inches × 48 inches × 24 inches high. A large square firebrick is placed above the hole, supported on bricks, in order to spread out the flame and keep it from impinging on the retort too much, and also serving to distribute the heat more evenly.

The products of combustion pass through the flue system, which consists of four horizontal circular flues approximately 15 inches deep × 9 inches broad built right round the retort, and so arranged that the gases must travel round the retort before passing to the next flue above. Each flue is provided with four explosion doors. From the topmost flue the gases descend to the waste gas flue leading to a 100 foot steel chimney stack.

The condenser platform consists in each house of an upper and lower deck, the upper deck serving to support the condensers and the fume main, and the lower deck to carry the acid mains. The receiver platform is built alongside the lower condenser deck, and a little below it in level, so that the main can be set with a good fall in the direction of flow. The condenser deck is built of wood or steel, and acid resisting brickwork. The receiver platform is supported on acid resisting brick piers.

Nitric-acid condensers.—The final arrangement of the Hart condensers was carried out as shown in Fig. 3, and gave the most satisfactory results.

The uptake from the retort is of 3 inch silicon-iron metal, and is carried up vertically about 4 feet before bending over to connect with

the cascade, which slopes down at an angle of 80° with the vertical. The cascade is built of straight pipes. Cascades of silicon-iron have been put in, also cascades of ceratherm. Both are an improvement on potteryware.

The gas from the retort passes through these 8 inch pipes into the end of an 8 inch pottery junction pipe E, the pipes being laid with a slight fall so that they drain into the junction pipe.

The other end of the junction pipe is bent downward and is connected to the acid mains in the manner hereafter described. From this junction pipe two stand pipes G are taken, each carrying a front condenser header K suitable for taking 26 glass tubes 1¾ inches outside diameter. The stand pipes G are moulded with flanges in order to support them on the condenser framing. The glass tubes above mentioned are connected to two back headers M of similar design to headers K except that the lower ends of these headers are closed whilst the tops are formed with open sockets. The two sets of headers are set at a level such that the glass tubes drain towards the front headers, the condensed acid passing thence into the junction pipe E. The gas passes through these two banks of tubes in parallel and thence into a 6 inch arch pipe N connecting the upper ends of the two back headers M.

From the centre of the arch pipe N another arch pipe O conducts the uncondensed gases into the top of a third back header M. This is exactly similar to the two back headers previously mentioned and is connected to a front header L by means of 26 glass tubes 1¾ inches outside diameter. This header L is similar to the front headers K except that it has an open socket at the top instead of being closed, in order to permit uncondensed gas to pass to the 9 inch fume-main through the 4-inch damper pipe Q and the 4 inch arch pipe R.

The tubes in this bank are set so that they drain towards the front header L. This header stands in a third stand pipe G exactly similar to those supporting the other two front headers, the acid condensed in the tubes passing down through the stand pipe into a sealed catch pot J.

The overflow from the catch pot is 1¼ inches diameter and is led to a 2 inch Y-pipe X, the other inlet to which is connected to the lower end of the junction pipe E, so that acid condensed in all three banks of tubes is led to the Y-pipe. Glass tubes V and W are inserted in each of the connections so that the flow of acid may be observed. The acid passes from the Y-pipe X into a still watcher marked Y. This consists of a 2 inch U-pipe formed with a socket at the inlet and with an overflow basin, 6 inches in diameter, on the outlet leg, the basin being fitted with a glass dome and two 2 inch discharge pipes. In the event of pressure rising in the retort the glass dome will lift and the pressure be relieved.

The discharge pipes from the still watcher are connected to the weak and strong acid mains as shown, with cocks in each connection, so that the acid discharged from the condensers may be diverted to the strong or weak main at will.

All joints in the pottery pipes are made with a cement of the following composition :—

						Parts.
Asbestos powder	-	-	-	-	-	40
Asbestos fibre	-	-	-	-	-	8
Tallow	-	-	-	-	-	2½
Whiting	-	-	-	-	-	20
Boiled linseed oil	-	-	-	-	-	21

As there are more than 150 joints connecting the condensing system of each retort, the greatest care is necessary in selecting jointing material. The glass tube is placed in position and a ring of ¼ inch blue asbestos cord put in. A layer of asbestos putty is then inserted, the same repeated, and the whole faced with a mixture of asbestos powder and silicate. When this is properly set the surface of the joint is painted over with bitumastic paint. These joints, if properly handled, will last quite a long time. Sudden pressures on the retort, and sudden rushes of gases, must be avoided if such joints are required to have a long life.

These tubes are covered by means of scouring cloth, which serves to distribute water evenly over the tubes. The cloth is torn up into strips 4 inches wide, and is interwoven with the tubes, round the first tube to the right, thence to the second tube by the left, and so on down the standard. The cloth is kept from the standards about 4 to 6 inches. Rubber rings, ½ inches in diameter, are used on the glass tubes at their lower end to keep water from the joints where the glass tubes enter the standards. The quantity of cloth required for a set of three standards is 300 square feet. About 2,000 gallons water are run down a set of tubes per charge. The surface area of the glass condensing system (three sets) is 150 square feet. The water from the condenser standards is collected in a trough 6 feet 6 inches × 4 inches deep and 6 inches wide, from which it is run down to a common drain fitted with a V-notch. Samples are taken daily and analysed. The average loss of acid is 10 lb. nitric acid per house per day.

Nitric acid mains.—The acid mains are 3 inches in diameter and are built of 2 feet to 2 feet 6 inches lengths of pottery pipe. They run the entire length of the house, and are supported on brick arches on a platform below the condensers. Each condenser delivers into the mains by branch pipes.

The strong main connects with a 3 inch pottery pipe which runs at the back of the strong receivers, and gives off branches to the three lead-cooling coils. Each branch is closed by means of a 3 inch pottery cock, and there are three more similar cocks along the main, behind the receivers, so that any particular section may be cut off for repair work without interfering with the running of the plant.

The weak main is continued along the platform and outside the house to the weak receivers. Eight branch pipes with suitable earthenware cocks connect to the eight receivers which are reserved for weak acid.

Three 2 inch lead coils are provided in each house. They are immersed in iron tanks containing running water. The acid exit from each coil branches to two lead receivers, a 2 inch pottery block cock being provided on each branch to enable the acid to be run to either or both receivers.

Nitric acid receivers.—The strong receivers are built of 20 lb. lead in the shape of cylinders, 8 feet 3 inches diameter × 30 inches deep, and each receiver has a capacity of about 6 tons of strong nitric acid. Six vertical iron T-beams round the circumference and two encircling iron bands strengthen the receiver, and the top is supported by six lead-covered iron I-shaped rails. A man lid and a 4 inch dipping hole are provided in the top of each receiver, the former being permanently bolted on, while the latter is covered by a lid which fits into a lute containing a little mineral jelly. The outlet cocks from the receivers are 2 inch pottery block-cocks, which connect with a 4 inch lead main, to the mixing station. Under each outlet cock is a lead drip tray, and these trays connect by a pottery pipe to a pottery blow egg. A 4 inch pottery pipe connects each receiver to the fume main, and a lead pipe perforated on the upper side passes round the interior of each receiver and serves for the introduction of compressed air to mix the acid before sampling.

Ten receivers are provided for weak acid. Two of these are reserved exclusively for tower acid. They are of pottery, of 100 gallons capacity, and of cylindrical shape. The lid is separate, and is jointed on with the usual asbestos jointing. The acid is run out by gravity through 1 inch pottery bib-cocks into a 3 inch pottery main to the mixing station. A 2 inch pipe connects each receiver to the fume main, and there is a dipping hole, closed by a pottery cover, in each lid. The receivers stand upon a flooring of acid-resisting tiles. Any drips from the cocks are collected in a pottery pipe placed underneath and led to a carboy placed beneath the stage.

Fume mains.—The exit from the third Hart unit of each condenser set is connected to a large 8 inch pottery fume main which passes along each house. A main of the same size branches off to the absorption towers from the middle of each house. To the first main is connected a smaller 6 inch main which serves to exhaust the receivers by means of the 4 inch and 2 inch branches above described. The fume main is built on a slight slope so that any condensed acid runs to the lowest point, where it is drawn off to the receivers through a suitable 2 inch pipe.

Nitric acid eggs.—Two earthenware eggs, each of 77 gallons capacity, are installed beneath the strong receiver platform, one in each house. Drip pipes and runs from all parts of the house, drip trays and pottery jugs are all emptied into these eggs. Any acid spilt from any cracked mains, and acid collected in carboys, is put into these eggs, and the contents blown to the receivers by compressed air at 10 lb. pressure. The acid is delivered through a 2 inch pottery line, to either strong or

weak receivers as required. The inlet cock is of pottery and the air pipe of lead. Connections are made by means of flanged joints packed with asbestos and compressed with iron clamps.

Absorption towers.—The 8 inch pottery mains already described above are carried outside the houses on wood trestles to the absorption towers. There are two sets, each of eight towers, one set being connected to each house (Fig. 4). The towers are elevated on brick piers 12 feet high. Both deal with the uncondensed gases from the houses. The towers are built of stoneware, and are constructed in five sections. Each section is 3 feet long × 3 feet diameter. The column of five sections rests on a pottery saucer 9 inches deep and having an internal diameter of 3 feet 6 inches. The bottom section has a 9 inch take-off to connect as gas inlet to the fume main. The towers are themselves packed with graded quartz, which rests on a tiled archway built in the saucer to give easy access for the fumes. The size of the quartz packing varies from 6 inch stuff at the bottom to 2 inch stuff at the top. At each section of the joints a 2 inch perforated plate is provided with 1 inch holes. Packing the absorption towers with lumps of quartz and also with Accrington pottery rings has been tried and it was found that the pottery rings had the following advantages over quartz packing : —

(1) Proportionally greater free space.
(2) Proportionally greater wet surface.
(3) Greater uniformity.
(4) Less liability of choking.
(5) Small lateral pressure on the tower body.

In the first record of this series on " Denitration of Spent Acids," particulars of the earthenware ring packing are given, and it is shown that in a total volume of tower of 99 cubic feet, 38 cubic feet are occupied by the packing leaving 61 cubic feet of free space. The total surface offered inside each tower is 2,111 square feet. The saucer at the foot of the tower is fitted with a 1 inch cock, which delivers the weak acid into a constant level receiver situated just below. At the top of each tower is a pottery acid distributing bottle known as a beehive, fitted with four ¾ inch bib-cocks. Two of these are connected to deliver acid from the beehive down the tower of which it forms a part. The other two are provided so that acid may be forwarded through the same tower again, or part diverted to the next tower in the series through 1 inch glass pipes.

Constant level receivers, already mentioned, are made of pottery, and measure 27 inches in internal diameter. They hold acid to a depth of 20 inches, at which level each is connected with the next in the series by means of a 1¼ inch glass tube, which maintains a constant level in all receivers.

Pohle air lifts are used for raising the absorption acid from the constant level receivers to the tops of the towers. The gases conveyed along the fume main enter at the foot of No. 1 tower, are drawn through the tower, leave by an 8 inch U-bend, and are conveyed to the bottom of the second tower and so on through the series. The absorption is

carried out by counter-circulation of absorption liquid (water or weak nitric acid) and the nitrous fumes. A constant supply of acid to the well is received from the constant level receivers. Air at 15 lb. per square inch is supplied *via* the glass air tube, and a continuous but jerky lift of acid to the beehive on top of the tower is maintained.

. The compressed air required for operating the Pohle lifts is carried to the tower along a 2 inch branch pipe from the main air service at 80 lb. per square inch and is reduced before it is used at the towers to 15 lb. per square inch. It is connected to the individual lifts by means of stout rubber connections taken from a 1 inch pipe. The compressed air serves two purposes, viz., to elevate the acid from bottom to top of the tower, and also to oxidise the nitric oxide passing through the towers to nitrogen peroxide, which in contact with water under suitable conditions forms nitric acid.

A 30 inch diameter Sirocco fan, driven by a 10 h.p. motor, causes a blast of air to be driven through a pottery injector. The suction pipe, also of pottery, communicates with the gas outlet front No. 8 tower. By this means a vacuum of 6 inches on the water gauge is produced on No. 8 tower. This falls away to 2 inches on No. 1 tower, and to ⅛ to ¼ inch at the retort head. The ejector is perfectly simple, and causes no trouble. The fan delivers at a pressure of 6 inches water gauge at the rate of 40 cubic feet per second through a 9 inch pipe.

Nitre cake disposal.—This extends the full length of the house. The floor is built of acid-proof bricks laid on a concrete bed, the joints being run with molten sulphur. This floor is easily cleaned, and is very resistant to acid. Each retort is provided with an iron chute 9 feet long × 1 foot wide × 1 foot deep, the top end of which rests in the recess of the retort wall, around the tapping hole, and the lower end fits into a wooden extension chute of similar dimensions, except that it is about 12 feet long. A wooden trestle supports the whole. The lower end of the wooden chute rests over a lead-lined drain 12 inches deep × 18 inches wide. This launder runs the whole length of the retort house. Each retort is provided with a 2 inch water valve, situate at the junction of the water and wooden chute. This conveys a copious supply of water to dissolve the nitre cake down the wooden chute and into the gutter, whence it ultimately runs into the sea. This process was soon abandoned, and all nitre cake was recovered, in some cases in a granular form, and in others in lump form.

A portion of the nitre cake is recovered in granular form by directing the molten nitre cake along the chute to a point where it falls and is met by a powerful blast of compressed air from a 2 inch horizontal iron pipe, with a slit 4 inches long, adjustable in width from ¹⁄₅₀th of an inch to ¹⁄₆th of an inch. The molten nitre cake is immediately solidified and falls like fine snow on the floor, roof and sides of the blowing house, which is 40 feet long × 15 feet wide × 12 feet high. The material is in fine granular form, and finds a ready market. It is usually put up in 40 gallon casks. The present system of recovery is as follows :— The iron chutes from four retorts each connect with another chute running at right angles. From one end of this latter chute another projects downward

to the point where the molten nitre cake, after falling 13 inches, meets the air from the horizontal pipe at an angle of about 45°. The four adjacent retort chutes are similarly fitted. These two iron chutes terminate at the entrance of the blowing shed, and each chute is provided with a blower to itself. The pressure of air at this 2 inch pipe is 80 lb. per square inch, and a gauge is placed near by. When the pressure falls the cake is not properly granulated. The house to which the granulated cake is blown is built up of old galvanised sheeting, the floor being laid with acid-proof bricks laid in cement and jointed with asphalt.

Another method of preparing the nitre cake in granular form consists in running it from the retort into a circular iron pan in which blades or ploughs attached to radial arms are revolving (Figs. 5 and 6). The constant agitation prevents the nitre cake setting as a whole, and by constantly presenting new surfaces to the air a rapid cooling effect is brought about leading to the formation of granules, and these in turn by grinding upon one another eventually suffer reduction to powder. There is a discharging door at one side of the pan and the ploughs are placed at such an angle that when the door is opened the powdered nitre cake is gradually discharged from the pan.

The whole of the North house at Gretna is provided with ordinary pans, four to each retort, size 12 feet × 5 feet × 8 inches. These pans rest on brickwork, and are raised some 3 feet from the ground. A railway track passes along the line of pans the full length of the house. The charge of molten cake is run evenly into the four pans allowed to solidify, and then broken up with a hammer and thrown into the wagon alongside.

The only differences worthy of mention from the description of the South house tapping floor (where the granulation process is used) are as follows :—

(1) Floor constructed of concrete, which does not resist the action of acid as well as the acid-proof bricks.

(2) A half-rounded pottery drain takes the place of the leaden launder in the South house.

Water supply. —This is similarly arranged in both houses. A 4 inch main in each house, fitted with a main stop valve, supplies water to the Hart condensers. The outer 4 inch main with stop valve supplies water to the tapping chutes. This main completes a circuit right round the house, branches being taken off at intervals for the following purposes :

(1) For tapping retorts.

(2) For 1 inch hose pipe connections.

(3) For cooling worms for strong receivers, &c.

With a view to effecting economies in the water consumption on the plant, large galvanised tanks were placed on girders about 15 feet from the ground in both houses. These tanks receive waste water from the acid mixer coils, which previously ran to waste. Each tank holds 1,000 gallons water. The outlet pipe from the tank connects the 2 inch main supplying the water for tapping the retorts, and by means of a ball valve arrangement (similar to ordinary cistern), a plentiful supply

Of water can always be depended upon, either from the mixers overflow or from the main.

Joints for nitric acid work.—The method used for joining pottery socket-pipes, &c., for both liquid and fume is as follows :—

A ring of blue asbestos cord of suitable thickness is first placed in the joint, then the socket is filled up with grease packing

					Parts
Asbestos powder	-	-	-	-	40
Asbestos fibre	-	-	-	-	8
Tallow	-	-	-	-	$2\frac{1}{2}$
Whiting	-	-	-	-	20
Boiled linseed oil	-	-	-	-	21

almost to the top of the socket, and the joint finished off by pointing with silicate packing. Where the socket is deep enough, two rings of asbestos cord are put in with a layer of grease packing between each.

The men employed luting socket pipes are very apt to hammer in a thick cord with the idea of getting a tighter joint, but this should be prohibited, as the cord swells and the socket is split. This applies more particularly to pipes, &c. made of special material, e.g., ceratherm, &c.

Glass tube joints on Hart condensers are made in a similar manner, but in every case two rings of cord must be used in order to make a tight joint.

On some plants the chemists in charge prefer the joints to be tarred when finished, others like them painted with weak nitric acid, but if water can be kept away from the joint (as it ought to be) painting with a fairly strong solution of silicate of soda puts a fine hard finish on the joint, and prevents the jointing material cracking. Painting with nitric acid helps the joints to harden quickly, but does more harm than good to the luting.

PLANT OPERATION

Sampling of nitre.—The crude nitre is sampled weekly. When sampling, about 20 cwts. are usually taken down, halved and quartered in the usual way until 28 lb. are obtained. This is then submitted to the laboratory. The following is an average analysis of the sodium nitrate :—

			Per cent.	
$NaNO_3$	-	-	95·85	$\left.\right\}$ $NaNO_3$ 96·6 per cent.
KNO_3	-	-	0·98	
$KClO_4$	-	-	0·14	
NaCl	-	-	0·50	
Na_2SO_4	-	-	0·20	
Insoluble	-	-	0·06	
Moisture	-	-	2·00	
Iodates	-	-	Trace.	

The nitre is usually dried so that the finished sample contains $\frac{1}{2}$ per cent. moisture. This brings the nitrate content of the sample about 98 per cent.

Usage of sulphuric acid.—In each report two tons of nitre are distilled with approximately two tons of 92 per cent. sulphuric acid. The following are actual figures :—

2 tons nitre at 98 per cent. $=$ 1·96 tons of pure salt.
2 tons of sulphuric acid at 92 per cent. :—

    1 inch dip in tank $= 0·059$ ton.
    34·5 inch used $= 34·5 \times 0·059 = 2·035$ tons at 92 per cent.
                  $= 1·872$ tons of H_2SO_4.

Ratio of sulphuric acid to nitrate in terms of pure chemicals

$$= \frac{H_2SO_4}{NaNO_3} = \frac{1·872}{1·960} = \frac{0·956}{1·000}.$$

Firing the retort and gas consumption.—The above materials are put into the retorts, the lid is luted down with cement, and the gas fire lighted. The damper is adjusted at the back of the flue, and the air ports in the front, to give complete combustion of the fuel. The following are average analyses of the producer and waste flue gases :—

	Producer gas.			Waste flue gas.
	Per cent.			Per cent.
CO_2 - - -	4·0	CO_2 - -	8·1
CO - - -	28·0	O_2 - -	9·9
H_2 - - -	11·1	N_2 - -	82·0
CH_4 - - -	1·4		
N_2 - - -	55·5		100·0
	100·0			

In order to find out the exact gas consumption, a rotary meter was fixed on to retort 33. Experiments were made under varying conditions to find out exactly what the coal consumption was. The average figure recorded was 30,000 cubic feet reduced to normal temperature. This is for a charge of 2 tons nitre, which gives 1·43 tons of HNO_3.

    1 ton of coal gives 130,000 cubic feet of gas,
    1 charge (*i.e.* 1·43 tons) uses 30,000 cubic feet of gas,
    ∴ 1 ton uses 20,000 cubic feet of gas,
or equal to 0·154 tons of fuel per ton of HNO_3.

Distillation.—When a retort is tapped, the charging lid is removed and the retort dipped to make sure it is tapped clean. It is a rule on the plant that the tapping hole must be plugged before the nitre is charged; this is done to avoid the possibility of the acid being run in before plugging.

The nitre charge of 2 tons is weighed in four bogies, 10 cwts. each, and is delivered into the retort by means of a portable chute. After tipping, the bogies are swept clean, and any nitre spilt is swept up and added to the retort. The charging chute is now removed. The seat for the lid is covered with clay, the charging lid put on and cemented down

with ordinary cement. In running in the acid by the first method the centre plug is left out, but in the second method the plug is luted down with clay and cement.

Meanwhile the charge of sulphuric acid, consisting of 2·05 tons of 92 per cent. acid, or 2·01 tons 93 per cent. acid, is measured into the feed tank. There are two methods of running in the acid. In the first method a temporary connection is made from the tank to the retort, using a short piece of iron pipe, which dips into the acid charging hole in the lid. In the second method a permanent connection from the tank to the retort is used, which is independent of the charging lid. A cast-iron tee-piece is bolted to the retort cover, and is connected to the pipe from the tank by a 2 inch iron pipe with a U-bend to minimise the effects of a blow back whilst charging. The tee-piece on the retort is necessary, because the acid inlet must be cleaned after practically every charge.

After the acid has been added and the plug luted down, the fire is started, unless the retort be very hot, in which case it is advisable to wait a short time. Sufficient fire will be put on to make distillation commence in about one hour. If the retort be cold, a much longer time will be required, viz., up to three or four hours.

For ordinary running, a 14 to 16 hour cycle is the most usual, with a distillation time of 12 to 14 hours. As soon as a retort is warm, a few brown fumes appear in the condenser tubes. A clear indication of the commencement of distillation is given by a rapid rise in the uptake temperature to 90° C. When the pot temperature has reached 90°–110° C., the temperature will steadily rise for the next two or two and a half hours, with a regular evolution of nitric acid vapours. As soon as the " pot " reaches a temperature of 115°–120° C. the reaction between the sodium nitrate and the sulphuric acid becomes violent. The pressure in the retort rises, and the manometer on the retort head gives indication of this. At this time it is usual to put out the fire while this violent reaction is in progress to avoid damage to the pottery. This reaction lasts from 5 20 minutes, depending largely on the rate at which the pot temperature has been raised. This period is very clearly shown in the condensers. Before it occurs the retort distils regularly and the gases in the condensers are brown, but during this period the vapours in the condensers are colourless, or almost so. As soon as this violent stage of reaction is over the tubes quickly become colourless again, and the fire is re-started.

During the height of the reaction the pressure in the cascade is shown as 3-4 inch water pressure by the manometer. Whenever the manometer shows less than 1 inch pressure it is safe to light the fire. For the next four or five hours the rate of distillation drops off and the uptake temperature will drop somewhat, from 135° to 130° C., and the fire must be slightly increased.

From now onwards the specific gravity of the liquid distilling is taken every hour by the " reaction man." When the specific gravity of the distillate falls to 1·465 the acid made is run to the weak acid receivers; up to this point it has been running into the strong receivers.

The distillation is now steadily continued until. the specific gravity of the distillate reaches 1·340; then the fire is extinguished, and the retort is ready for tapping. A retort is not actually ready at 1·340, but there is sufficient heat in the charge to finish the distillation while preparations are made for tapping. The end point is also shown in the condensers, because very little distillate is coming over. The uptake temperature drops considerably towards the end.

The term "cycle" means the time which elapses between the charging and emptying of a retort. "Dead time" means the length of time between the pot being empty and again charged. The cycle aimed for here is one of from 14–15 hours, which generally means a distillation period of 10 11 hours. "Blows" are very uncommon with the above times. With a lower cycle, say, 10–11 hours, in addition to breakage of pottery, there is an increased decomposition of the nitrate acid in the retort, which has been proved by the analysis of the gases in the fume main. Generally speaking, quick cycles do not pay. Longer cycles than 14–15 hours are not desired, as coal consumption goes up, and a larger quantity of nitrous acid is found in the distillate.

Tapping.—The tapping of the retort has already been described. Great care is necessary to see that the tapping hole is kept clean

When a retort is tapped two samples are taken of about 7 lb. weight. These are sent to the laboratory, and an analysis made for sulphuric content and nitric acid content. An average figure is as follows : –

> 28 29 per cent. total acidity as H_2SO_4.
> 0·06 per cent. HNO_3.

The acidity of the sample depends somewhat on the stage of tapping, and samples taken at intervals during a tapping gave the following results :—

> Tap 1—32·3 per cent. sulphuric acid ⎫
> Tap 2—27·6   ,,   ,,   ,, ⎬ Average 28·8 per cent.
> Tap 3—27·6   ,,   ,,   ,, ⎮
> Tap 4—27·8   ,,   ,,   ,, ⎭

Condensation.—During the process of distillation, the bulk of the acid is condensed in the first two headers of Hart tubes. A small quantity condenses in the third set of tubes, and this usually contains upwards of 5 per cent. nitrous acid. Uncondensed gases pass to the fume main and to the absorption towers. For efficient condensation there must be provided both a large cooling surface and free passage for the gases without restriction of any kind, otherwise the gases will pass through and escape condensation in the one case, or a "choke" will develop in the other.

It is necessary for proper working to have a good vacuum on the plant. If a retort is not in use the damper is put in the fume pipe so as to avoid any unnecessary air getting into the system. It is found that, for efficient condensation, a quantity of 1,500 gallons of water should be passed over the condensers. This represents a consumption of 1,000 gallons per ton of nitric acid produced. Careful experiments

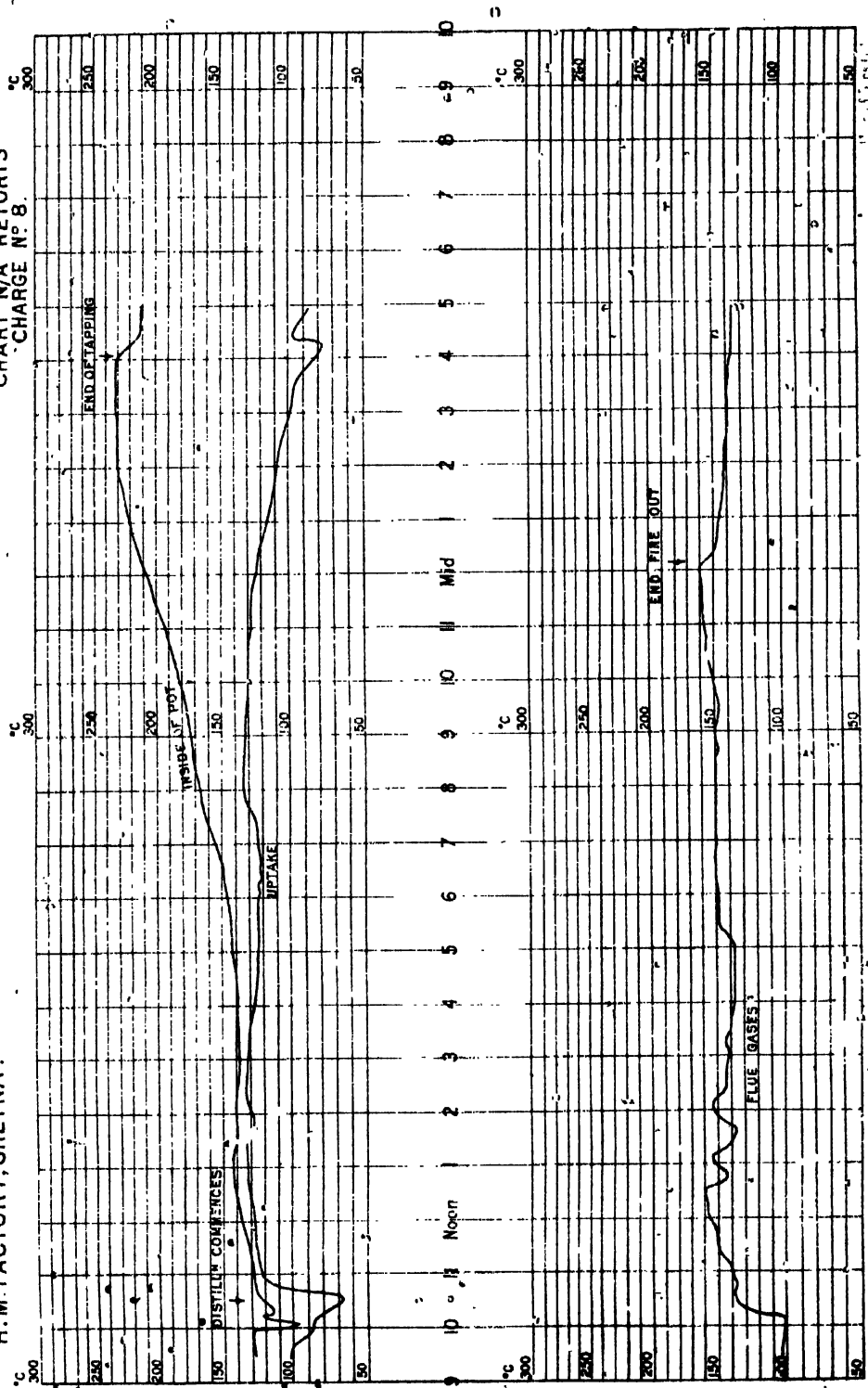H.M. FACTORY, GRETNA.

CHART N/A RETORTS
CHARGE Nº 8.

END OF TAPPING

DISTILLN COMMENCES

INSIDE OF POT

UPTAKE

Noon

Mid

END FIRE OUT

FLUE GASES

FIG. 7.

have been carried out to see how much nitric acid is lost from condenser joints. Twenty distillations were carried out in one retort. At the end of the twentieth, the water was only very slightly acid. The water was collected from each running, and up to the tenth, no acidity was shown.

Absorption.—Cold water is fed into No. 8 tower (the one nearest the exhaust), and the $\frac{3}{4}$ inch cocks on the beehive so adjusted as to give a progressive movement of acid through the system. Acid of between 60 and 65 per cent. strength is run off at No. 1 tower through a still-watcher, on which a hydrometer is placed, so that the strength of the acid produced may be readily observed. The acid run off from the towers is conveyed to the storage by means of a 2 inch pipe.

Analysis of acids made.— When a receiver is filled with acid, either weak or strong, a sample is taken and submitted for analysis. The analysis is returned shewing nitric and nitrous contents and specific gravity. Before sampling strong acid, compressed air, agitation is turned on so as to get an average sample. The average strength of the strong acid is in the neighbourhood of 92·5 per cent., with a nitrous acid content of 0·5 per cent. The weak acid is of average strength, 65-68 per cent. HNO_3, the nitrous content being almost nil. The acid recovered from the towers is usually 50 60 per cent. HNO_3, and the nitrous content 0·1 per cent.

Capacity of plant. –Assuming 12 hours as the lowest cycle consistent with efficient condensation and few breakages, the yield is 1·40 tons of HNO_3 from a charge in 12 hours. Allowing two hours dead time, the balance of the working day of 24 hours will give 1·16 tons of nitric acid.

1·4 plus 1·16 = 2·56 tons HNO_3 per retort per 24 hours.

Each house contains 24 units, two of which are generally allowed off for repairs.

∴ Total yield of acid = (24 – 2) × 2 × 2·56 = 115 tons HNO_3 per day.

Temperature charts. –Experiments were made using platinum resist-ance pyrometers and a Cambridge Scientific Instrument Company's recording machine. One of the pyrometers was encased in an iron tube and put inside the retort right down to the bottom.

No. 2 was put in the uptake of the retort, where the thermometer is usually put.

No. 3 was put at the back of the retort, where the gas flues enter the downtake. On reference to the accompanying chart (Fig. 7) it will be seen that the graphs correspond fairly well with the description in the text.

In the case of the uptake, when distillation starts the line is almost perpendicular. The dip on the line at two o'clock represents reaction time, when the fire is lowered, and as the distillation proceeds to a finish the temperature in the uptake falls.

The temperature inside the pot steadily increases from the start, and does not fall till the pot is empty of its nitre cake. It should be noticed that when the fire is put out the temperature continues to rise.

Working results.—Below is an extract from the working report for February, 1918, which shows in detail the working results. The efficiency of the plant lies between 96 and 97 per cent.; the quantity of H_2SO_4 used per ton HNO_3 produced is about 1·35 tons; and the quantity of $NaNO_3$ used per ton HNO_3 produced is about 1·39 tons.

The following is an average percentage return :—

Recovered as strong acid - - - - - 87·2 per cent.
　　　　　　　　　　strength = 92·5 per cent. HNO_3.
Recovered as weak acid (ex towers) - - - 10·4 per cent.
　　　　　　　　　　strength = 63·5 per cent. HNO_3.
Recovered as tower acid, weak - - - - 2·4 per cent.
　　　　　　　　　　strength = 56·5 per cent. HNO_3.

NITRIC ACID RETORTS

(North and South Houses.)

Monthly report —Five weeks ended 7.30 a.m. 2nd March 1918.

All weights in tons of 2,240 lb.

Total number of retorts charged —
　138¼ wet nitre; 1,359¾ dry nitre =1,498.
Total number of retorts tapped —1,505.

Charged.

Sodium nitrate.	Tons.	Per cent.	Tons.
Amount wet	276·5 $NaNO_3$	96·6 =	197·97 HNO_3
Amount dry	2,719·5 $NaNO_3$	98·41=	1,983·58 HNO_3

Sulphuric acid.

Amount	- 3,095·52 H_2SO_4	92·0 —	2,849·44 H_2SO_4
Coal used in			
producers -	401·54		

Nitric acid produced.

	Tons TNA.*		Tons TNA.
Amount strong	1,986·07 as HNO_3	92·4 =	1,835·62 as HNO_3
Amount weak	380·19 as HNO_3	63·75=	242·38 as HNO_3
Ex towers -	106·67 as HNO_3	59·7 =	63·67 as HNO_3
	2,472·93		2,141·67

Nitric acid. *Lost.*

Total TNA as HNO_3 charged to plant as $NaNO_3$ 2,181·55 tons.
Total TNA as HNO_3 recovered - - - 2,141·57 tons.
　　Unaccounted for at mixers - - - 20·71 tons.
　　　　Total amount lost - - 60·59 tons.
　　　　　　　　　　　　　　　　　(2·78 per cent.)

* Total nitrogen acids.

Physical Data.

Time of distillation—11 hours 43 minutes.
Time of cycle—14 hours 48 minutes.

Nitre cake.

Percentage free H_2SO_4—28·43 per cent. by analysis: 31·45 per cent. by calculation.

Fuel.

Coal used per ton of acid recovered	-	-	0·16 ton.
Coal used per ton of HNO_3	-	-	0·19 ton.

Nitric acid.

Average strength of acid recovered	-	-	86·6 per cent.
Tons produced per retort -	-	-	1·6 tons.
Percentage recovered as strong acid	-	-	85·7 per cent.
Percentage recovered as weak acid	:	-	11·3 per cent.
Percentage of weak acid recovered in towers	-	3·0 per cent.
Total recovery -	-	-	-	97·22 per cent.

From page 19 to page 57 the results are recorded of some of the investigations and trials which were carried out in order to ascertain the best way of running the plant with regard to efficiency and economy. These investigations were of the greatest assistance to the staff and led to the high efficiency which was ultimately attained on all the plants. They indicate the manner in which technical operations and problems should be attacked and studied.

STANDARDISATION OF RETORTS

Experiments carried out at H.M. Factory, Queen's Ferry, using Silica Condensers.)

Object of experiments.—These experiments were carried out with a view to determining the most suitable method of working the retorts in order to produce a maximum amount of strong acid and a minimum of weak using as small a quantity of fuel as possible, and at the same time working off the retort in a time as short as is consistent with these conditions.

Arrangement of retort. The retort was fitted up so that the acid could be run into carboys. One branch of the pipe was connected with a lead cooling coil immersed in water. This cooled the acid so well that it was at the temperature of the cooling water when it came out, and consequently fumed very little. The other was connected with an acid run, 10 feet long, made of small pottery pipes, and the acid flowing through this was cooled by water flowing on to the pipe. This did not cool very efficiently, but the acid came out cool enough not to crack the carboy and without undue fume. When the acid became weak it was turned into this run thus saving the lead coil. Otherwise, the arrangement of the retort was the same as in ordinary working.

x 16002

Method of working.—The carboys were changed at regular intervals of time, or, in some cases, when they were filled. They were weighed empty, and with the acid, and a sample of the acid of each carboy was taken. The temperature in the uptake from the retort was taken every quarter of an hour, or more often in special circumstances. The pressure in the uptake was also taken every quarter of an hour by means of a water manometer. A nitric acid trap, in the shape of a bottle, was put between the uptake and the manometer, so that any nitric acid coming through condensed here, and the water in the manometer remained pure and consequently of the same specific gravity. In three of the five experiments resistance pyrometers were put inside the retort and in the water-gas flue. The former was protected by a silica tube and a wrapping of asbestos soaked in silicate of soda while the leads to the top of the retort were encased in an iron tube. The temperatures given by the pyrometers were recorded on a continuously recording instrument. All alterations in the height of the fire, &c. were carefully noted.

All the retorts were gas-fired.

Results of Five Trials.

No. 1.—In this trial the retort was very cool to begin with, and hence took more than the usual time to heat up. A good fire was put on, and it took two hours to start to make acid. The fire was left unchanged, and a steady stream of acid continued to come through until three hours after it began to make. At this time the manometer, which had been indicating about ½ inch of suction, went down until it started to register pressure. The fire was turned out, and the top dampers put up. The positive pressure rose to about 1½ inches and continued for about ¾ hour, but during this time no readings of temperature could be taken because the thermometer had been cracked at the beginning of the blow. When the manometer again indicated suction the fire was lighted and turned up at intervals until two hours after the blow was over it was again a good fire and continued at this height until the end of the distillation. One hour and 10 minutes before tapping, the manometer oscillated violently for about 10 minutes.

No. 2.—In this case the retort was much warmer to begin with, and it started to make acid 1 hour 25 minutes after the fire had been lighted. A slight positive pressure developed at the same time as it started to make acid, the fire was turned low, but after about 10 minutes' suction was again registered on the manometer, and a moderate fire was put on. Two and a half hours after the distillation commenced a positive pressure of over 2 inches developed, and the fume began to blow out at the lid. The fire was put out and the fire-door and top dampers put up. The blow lasted ¾ hour. After it had ceased a fire was lighted, and this was soon raised until there was a good fire on. One and a half hours after the blow had ceased, a positive pressure again

developed, but lasted only a few minutes. The fire was turned low while it lasted. After another one and a half hours, another short blow occurred. When this was over the fire was turned up until the retort was tapped.

No. 3.—This was taken slowly with smaller fires than in the other cases. The period between the lighting of the fire and the beginning of distillation was 2 hours 45 minutes. This fire was moderate, and when distillation commenced it was checked, and a low fire kept until the blow started 3 hours 20 minutes after it began to make. The blow was a very slight and short one. After it a small fire was put on and raised to moderate heat only near the end. As in No. 1 there was violent oscillation of the manometer near the end of the distillation.

No. 4.—This was run through quickly to contrast it with No. 3. The retort was hotter to begin with than in previous cases. The distillation began one hour after the fire was lit, and it began to blow 2 hours 50 minutes after distillation started. A good fire was kept under the retort whenever possible. The blow was violent and lasted about 40 minutes. The water was blown right out of the manometer. Near the end of the distillation oscillation of the manometer was observed as in Nos. 1 and 3.

No. 5.—This one was run through fairly slowly. Distillation began after 1 hour 55 minutes. In this case there was no blow until 5 hours after it began to make, and the blow was very short and sharp, lasting only a few minutes and developing over 6 inches of pressure. After this the distillation went on steadily. One hour before the fire was put out slight oscillation of the manometer was observed. The fire was put out half an hour before tapping.

Temperature in the uptake.—The thermometer in the uptake gives the first indication of when the retort begins to make. The temperature rises in a few minutes probably 30° or 40° C. It will be seen by consulting Fig. 8 that distillation commences at different temperatures, varying, in these experiments, from 82° to 106° C.

But the temperature at which the gas distils off from the mixture of sulphuric acid and nitre must be nearly the same in all cases, so one is led to the conclusion that the temperature of the gases in the uptake is controlled very largely by the temperature of the upper parts of the retorts. Thus in distillation No. 1, although it had as good a fire under it as in any other distillation, the curve on the graph (Fig. 8) in the earlier parts of the distillation is well below the curves of Nos. 2 and 4. This is, no doubt, due to the fact that the retort and brickwork were quite cold to begin with, and the upper parts of the retort would take a long time to heat up. The contrast between Nos. 2, 4 and 3, 5 is particularly strong. Nos. 2 and 4 started with a good fire, and here the gas came over at temperature well over 100° C., while in Nos. 3 and 5 after distillation had commenced a small fire was used and the gas came over for a couple of hours at under 100° C.

In one respect the experiments, with the exception of No. 4, were not made under ordinary working conditions. It was necessary to leave the retort for a long period between each trial so that it cooled down more than under ordinary conditions, although it was kept as warm as possible by having all the doors closed. Nos. 3 and 4 were worked off without this rest between them, so that No. 4 will resemble more nearly ordinary working conditions in this respect, than the others. With ordinary conditions the upper parts of the retort and brickwork would be warmer, and the gas would be at a higher temperature to begin with than is shown in the curves. It will be seen from the curves that the thermometer gives no indication of the proximity of a blow though the temperature drops while the blow is in progress. The general tendency is shown in the next graph (Fig. 9), which gives a curve showing the mean of the five trials. The temperature rises fairly rapidly during the first two hours or more, then drops suddenly at the blowing period, subsequently rising slowly and steadily until near the end of the reaction, when it rises very rapidly. This last rapid rise is not shown in Fig. 9, but is clearly seen in each trial in Fig. 8.

The conclusion is that the thermometer in the uptake is of little use as an indication of the rate of distillation of the acid or as a warning of the proximity of a blow, and this is borne out by previous experience on the retorts. Its chief uses are to indicate the beginning of the distillation of the acid, and as a control on the temperature of the distilling gases so that these do not reach too high a temperature. The aim should be to keep the temperature as low as is consistent with the proper rate of distillation, so that as little of the acid is decomposed by the hot sides and top of the retort as possible. Starting with the retort always at the same temperature, the thermometer would be a good guide to the distillation, but, under ordinary conditions, to work always at the same temperature would be impossible, and an attempt to do so would lead to different rates of distillation.

Pressure in the uptake.—The pressure as indicated on the manometer was under ordinary conditions negative, that is, there was a suction in the uptake. This varied with the pull on the fume main, which itself varied considerably, according to whether there were any retorts open at the lid. The result was that the manometer readings when negative, had little significance except to indicate that the fan was operating properly, and that there was no blockage anywhere When the retort begins to make rapidly, at the blowing period the pull on the manometer gets less and less until a positive pressure is registered. The manometer gives the first indication of the proximity of the blow, and it is here that its value lies. It also indicates clearly when the blow is over and it is safe to light the fire again, and this is of special value, as it is here that time is frequently lost. Without the manometer there is no guide to the violence of the blow except the way in which the fume is blown out of the lid and other places, while with it the pressure developed is seen at once and the necessary precaution can be taken without any unnecessary cooling of the retort by opening
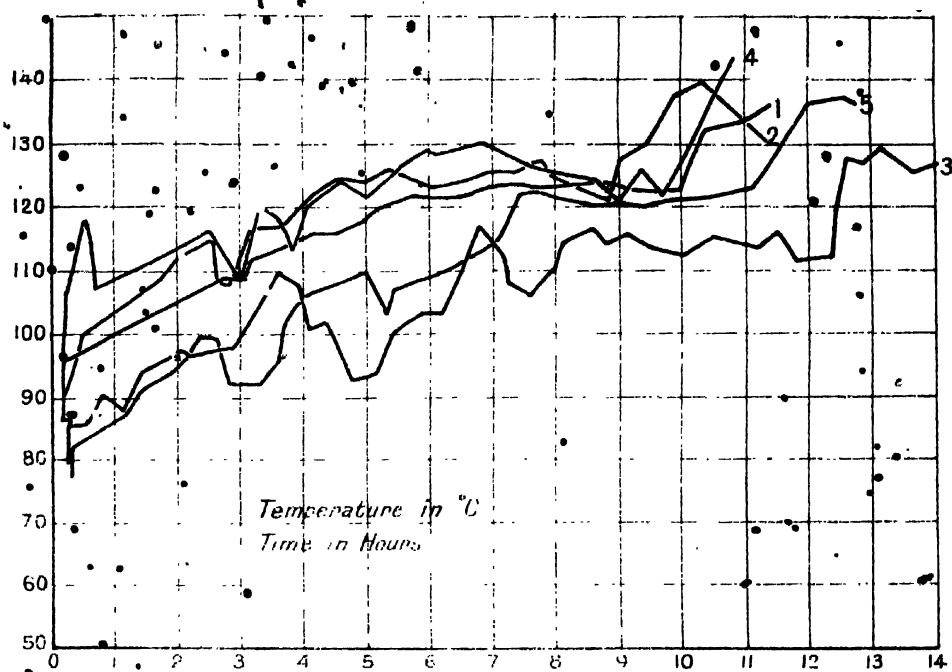
TEMPERATURE OF GASES IN UPTAKE.

FIG. 8

TEMPERATURE IN UPTAKE AND MAKE OF ACID.
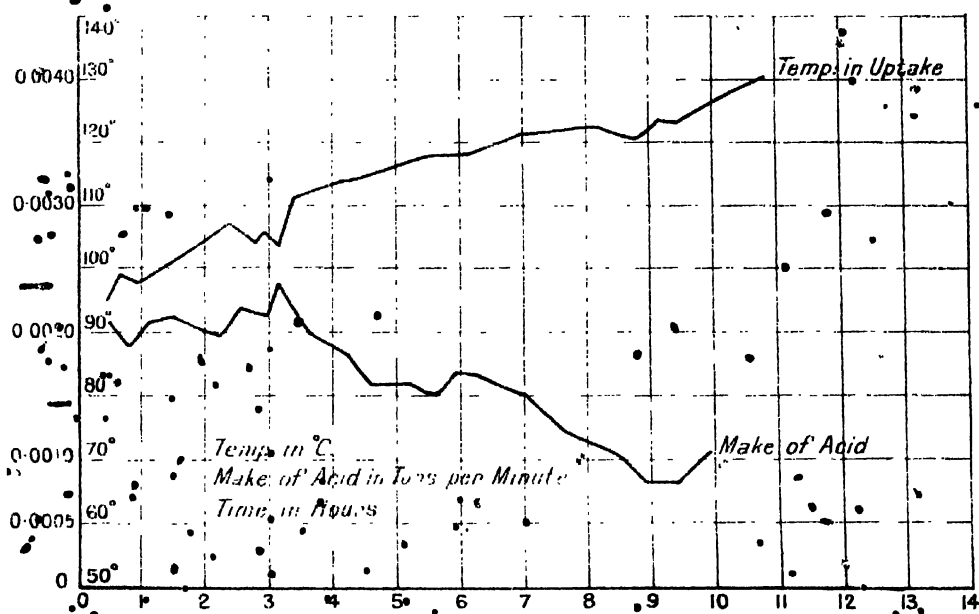### MEAN OF 5 TRIALS.

FIG. 9.

5 EXPERIMENTS

MAKE OF ACID ''
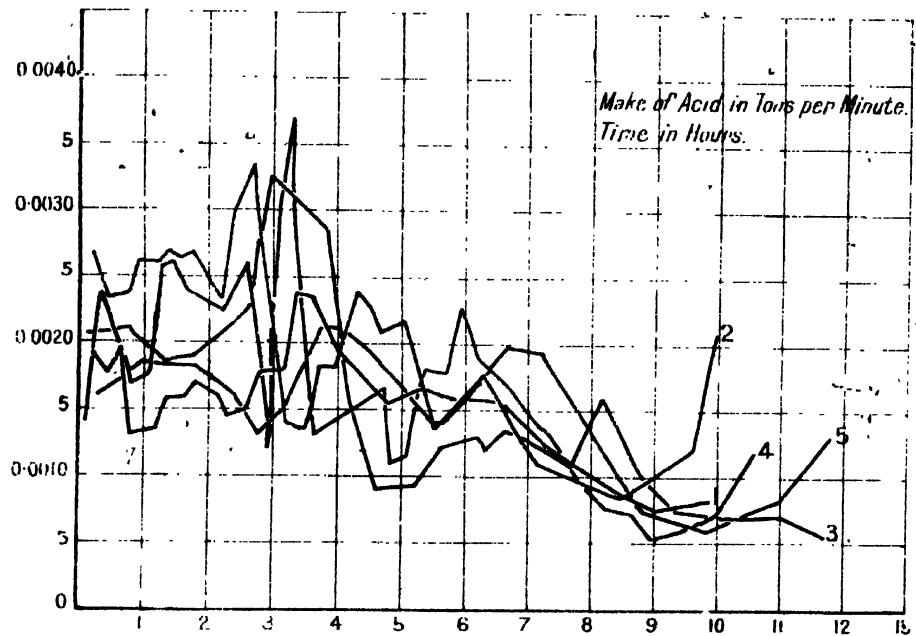
FIG. 10.

'damper, &c., a proceeding which is necessary only in severe blows, but which is frequently followed as a matter of safety in the absence of an indication of the probable violence of the blow.

Make of acid during distillation.—These experiments have shown very clearly that it is impossible, economically to have the make of acid even approximately equal all through the distillation. The curves in Fig. 10 show how in all the trials the rate of making acid varies, a great deal, while Fig. 9 shows how it falls off as the reaction proceeds. The retort begins to make rapidly and this rate is maintained for about 2¼ hours, during which time very little heat need be supplied. At this time the make usually drops a little, then suddenly increases enormously, and this sudden increase constitutes the blow. This phenomenon of very rapid making of acid at a period about three hours after distillation commences always occurs. If the retort has been pushed it occurs earlier than if it has not. In some cases (e.g. trial No. 5) it is delayed until much later than usual. This usually occurs when only a small fire is put on early in the distillation and when the retort is very carefully watched so that on any sign of blowing the fire is checked down. The blow is then usually of very short duration and rather violent. The following table shows the time between beginning of distillation and time of blowing and the quantity of acid condensed up to the time of blowing:—

	Lb.
Trial No. 1—3 hours - - - -	770
Trial No. 2—2 hours 30 minutes - -	852
Trial No. 3 —3 hours 20 minutes - -	880
Trial No. 4 - 2 hours 50 minutes - -	839
Trial No. 5 —5 hours - - - -	1,149

It will be seen that, except in a case of *No.* 5, the amounts of acid condensed are not very different in the different trials. The curves in Fig. 11 show that no sudden rise in temperature occurs in the contents of the retort at the blowing period. If the retort has been strongly heated during the preceding hours the blow is of a violent character, and to cool down the retort it is necessary to open all the explosion doors. Even then a great deal of fume is blown out of the lid or elsewhere with the attending loss of acid and other undesirable consequences. After the blow is over, the make of acid falls off even when a good fire is put on. To keep it up to its former height a very high fire would be necessary, and this would often result in a second blow. By keeping a good but not *very* high fire under the retort the rest of the distillation is usually steady. After falling off, the make again becomes fairly even, until two or three hours later it again begins to fall off slowly and continues to do so to the end of the distillation. It will be seen that at the end of the curves of *Nos.* 2, 4 and 5 in Fig. 10 there is an increase in make indicated. If the curves were continued they would fall again sharply, but the last carboy was left under the retort until all the acid had drained out, so that no figure could be obtained for it.

Relative quantities of strong and weak acids.—The results of the experiments clearly show the benefit to the relative quantities of strong and weak acid of working slowly.

' The following table shows the percentages of strong and weak acid, the ratio strong acid to weak acid (each reckoned as HNO_3) and the ratio strong acid to weak plus HNO_3 left in nitre cake. The percentages are reckoned on the total available HNO_3, taking the $NaNO_3$ in the nitre as 99 per cent.

Trial number.	Strong acid.	Weak acid.	Ratio $\dfrac{\text{Strong}}{\text{Weak}}$	Ratio $\dfrac{\text{Strong}}{\text{Weak}+N.}$*
	Per cent.	Per cent.		
1	90·2	1·9	47·5	39·?
2	86·7	6·4	13·6·	13·1
3	86·3	2·9	29·8	18·4
4	80·8	8·3	9·7	8·3
5	86·4	4·1	21·1	13·9

* N = HNO_3 in nitre cake.

In each case the strong acid is 92·8 per cent. strength.

It will be seen that *No.* 1 is the best. The sulphuric acid used in this case was 89·0 per cent. In several respects *No.* 1 trial differed from the others; although it had a good fire under it nearly all the time, the blow was only a mild one, the percentage yield of acid was high and the ratio strong : weak very good.

In the case of the other two which were driven fast, this ratio was distinctly not as good as in the case of the two driven slowly, as may be seen by comparing *Nos.* 2 and 4 with 3 and 5. It is probable that tho fact of the retort and brickwork being so cold to begin with in *No.* 1 is largely responsible for its unique behaviour, as this would prevent overheating of the sides of the retort. Comparing *Nos.* 3 and 4 the difference is particularly striking. These two were run especially for the purpose of comparing the strong and weak yields in the two cases of a retort run moderately slowly, and a retort pushed quickly. It so happened that the sulphuric acid used for *No.* 3 was only 87·8 per cent., whilst for *No.* 4 the strength was 91·2 per cent. This makes the comparison especially striking. The variation of the strength of the acid made with the time is shown in the results given at the end of the report. The acid at first is about 90 per cent., but rises quickly to 98 per cent., then after the first hour slowly falls until two or three hours before the end of the distillation, when it begins to fall rapidly.

Percentage recovery.—It will be seen that in every case this was high. Taking into account the HNO_2 in the acid, the percentage recovery in the five trials was as follows :—

Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
92·9	93·5	89·6	89·7	91·2

CURVES SHOWING TEMPERATURES OF CONTENTS OF RETORTS EXPTS 3, 4 & 5

TEMPORATURE IN RETORT (3.4.5.)

FIG. II.

Malby & Sons l'ith
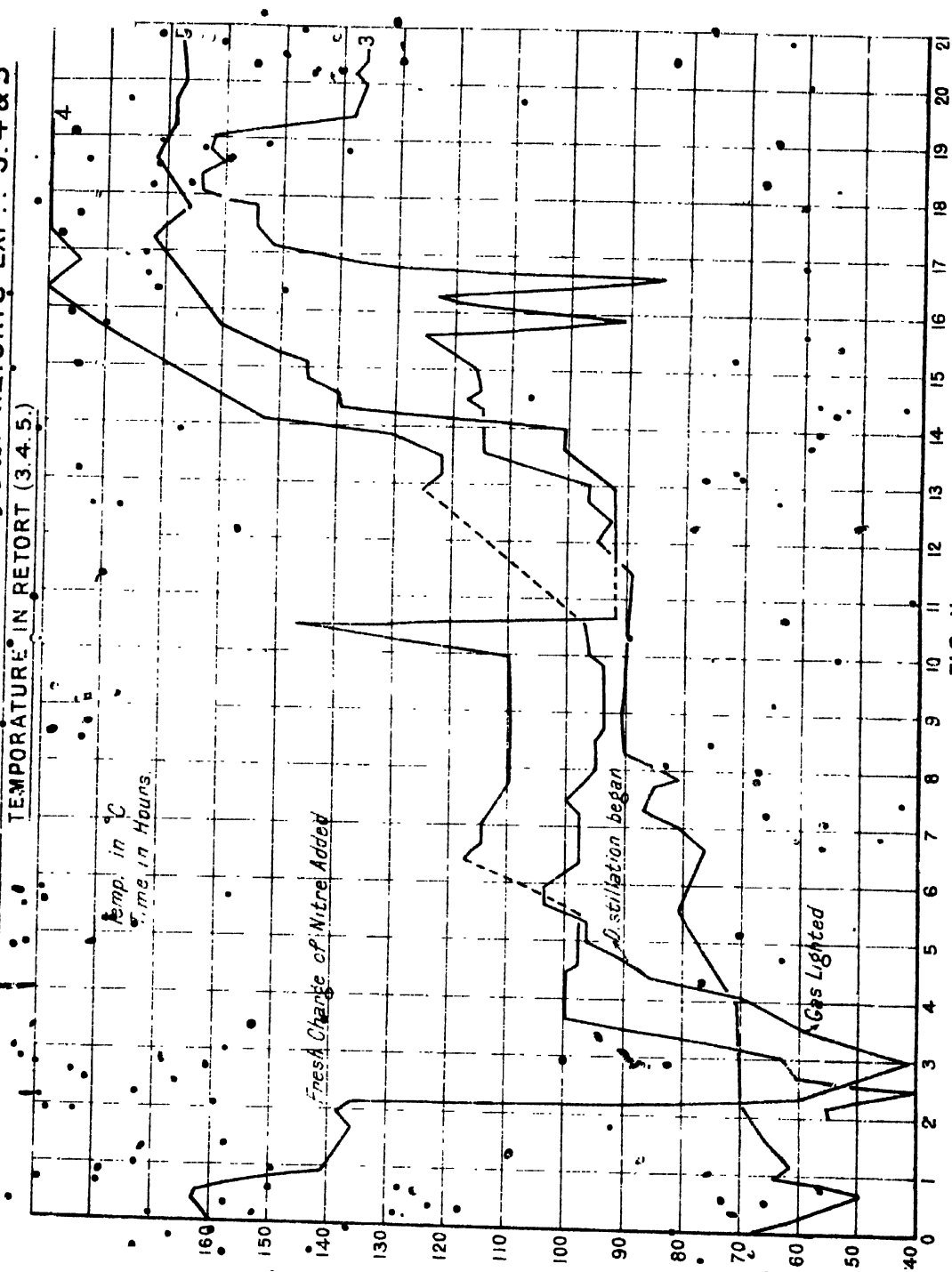
COMPARISON OF TEMPERATURES IN RETORT, IN UPTAKE & IN WASTE GAS, FROM FIRE.

TEMPERATURE IN RETORT, IN UPTAKE AND OF WASTE GASES FROM FIRE Nº 3.
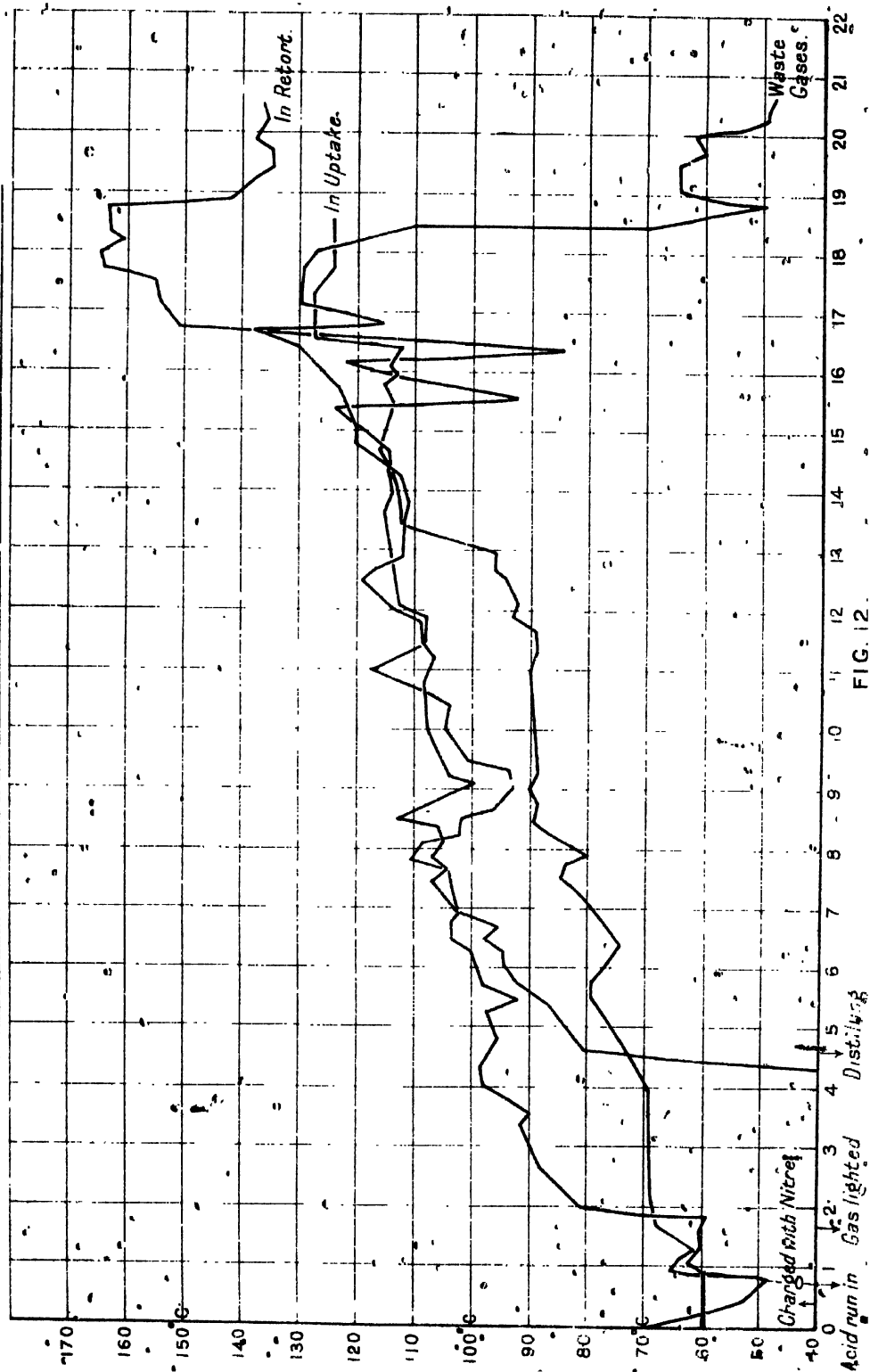
FIG. 12.

TEMPERATURE OF WASTE GASES FROM FIRE. EXPTS 3, 4 & 5.
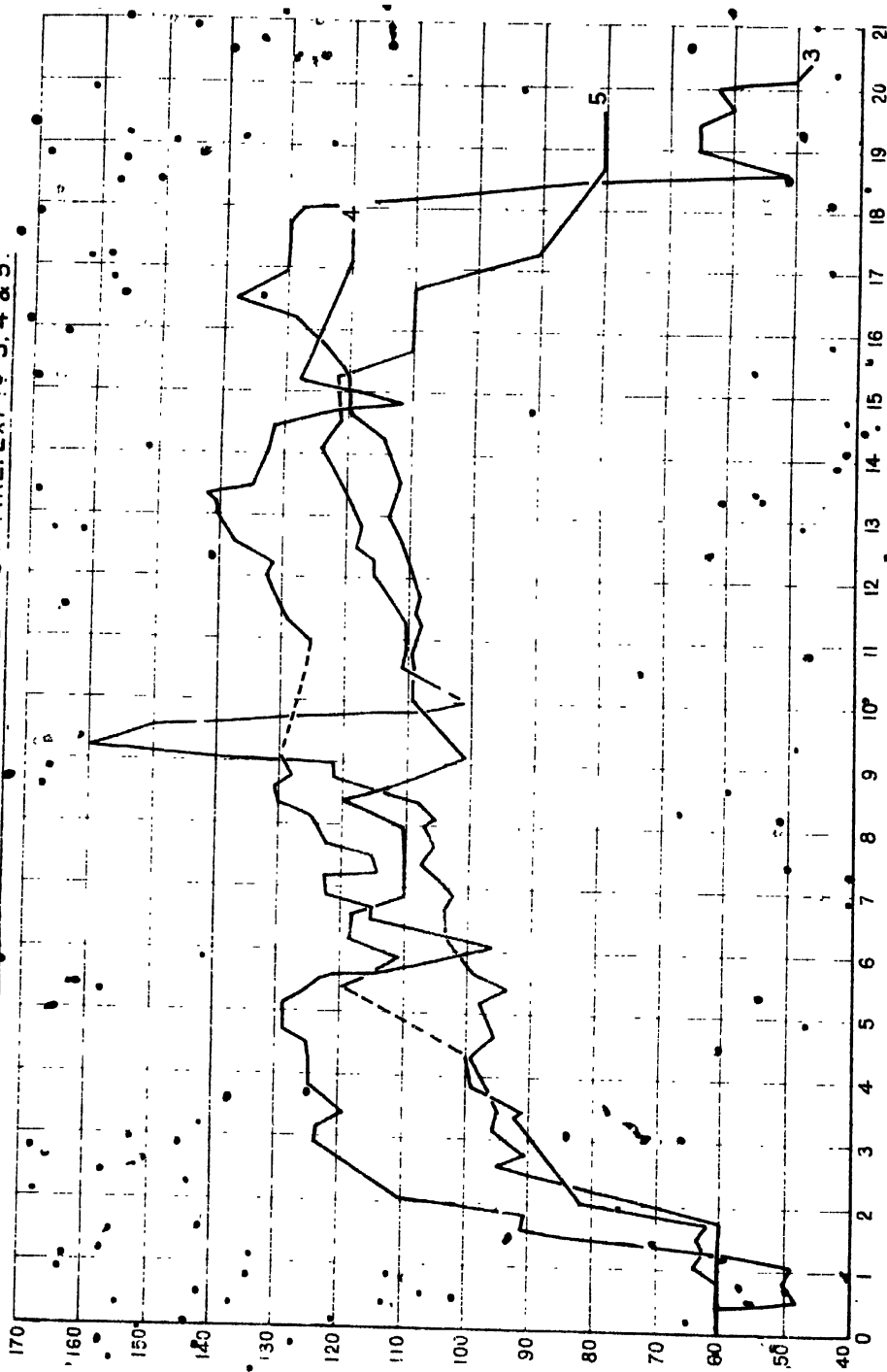
FIG. 13.

GAS CONSUMPTION Nºˢ 3 & 4.

Gas Consumption in Cubic Ft. per hr.
Time in hours.

The Areas enclosed by the dotted & solid
curves are proportional to the
quantities of fuel used in Nºˢ 3 & 4.
respectively.

FIG. 14.

3000

2000

1000

500

250

0

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21
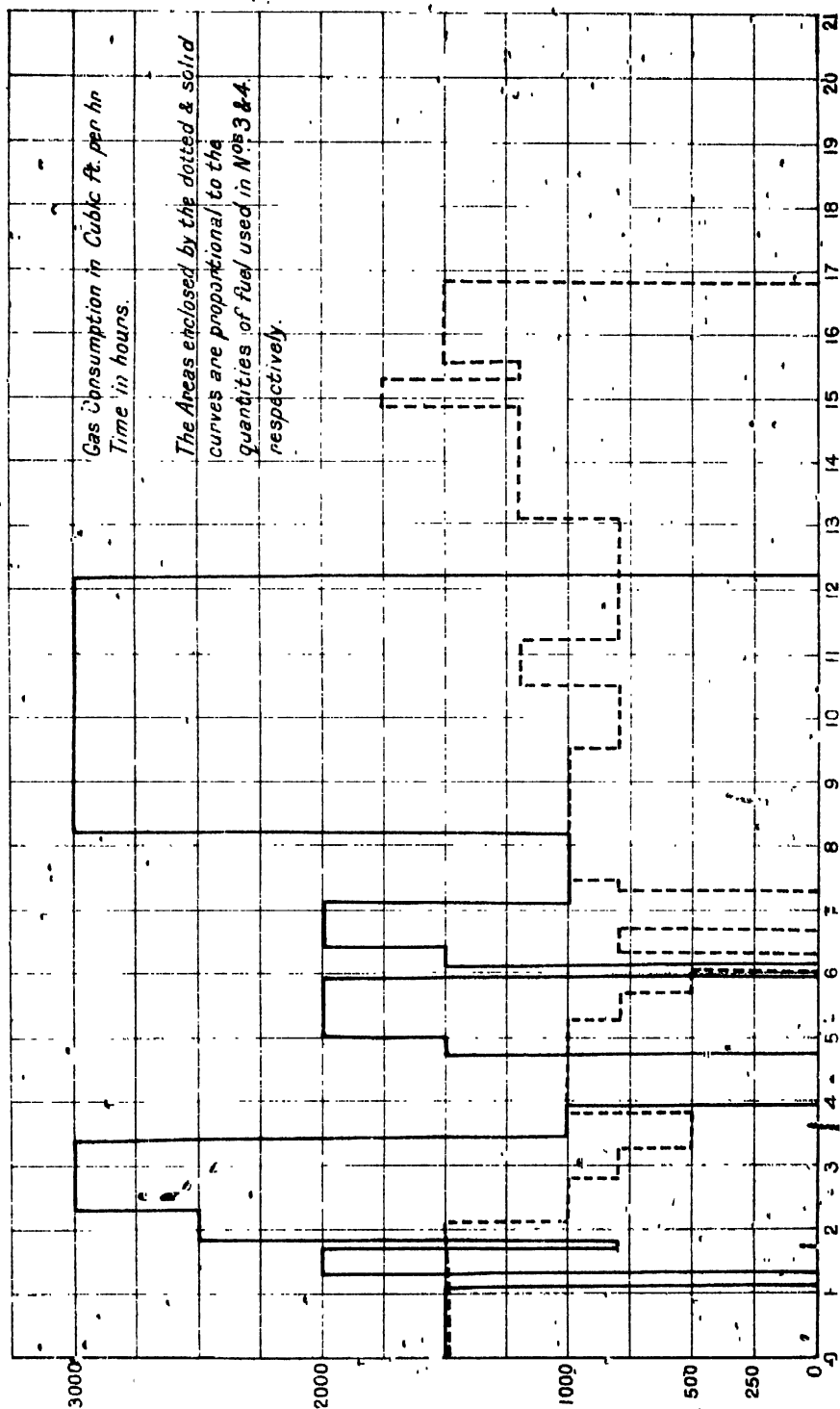
The mean of these is 91·2 per cent., so that only 8·8 per cent. of he total available acid was lost or absorbed in the absorption towers. It is difficult to understand why No. 3 should be the lowest of all when t was at the same time the one which was taken most gently.

Quantity of gas burned.—The firing of the retorts was carefully watched, and every alteration in the fire noted. The quantity of gas used was estimated, and this estimate is given in the following table :—

Trial number.	Time gas was alight.	Total gas used.	Cubic feet per hour.	Cwts. of coal.
2	10·2	23,000	2,250	4·6
3	15·6	19,000	1,200	3·7
4	11·0	24,000	2,200	4·7
5	13·8	23,000	1,650	4·6

(1 cwt. of coal is equivalent to 5,000 cubic feet of gas.)

It is seen that, in the case of the retort which was worked most slowly, the quantity of fuel used was the least although the gas was alight for the longest time. In the case of No. 5, which was worked as slowly as No. 3 at first, the gas was raised towards the end of the reaction with the result that it came off in less time, but used as much gas as Nos. 2 and 5.

Temperature of the mixture in the retort.—This was taken by a pyrometer as stated above. The results of trials Nos. 3, 4, and 5 are, shown in Fig. 11. In Nos. 3 and 4 the pyrometer was placed about 4 or 5 inches from the bottom of the retort, while in No. 5 it was higher up. From the appearance of the curve of No. 5 it seems probable that the pyrometer was sometimes out of the cake and sometimes in it. In No. 3 the distillation commenced when the pyrometer registered only 73° C. and continued for a considerable time with the temperature not much higher. It appears probable that in this case the mixture of nitre and acid was not uniformly heated all through, and that the distillation was taking place at the edges while the centre remained at a lower temperature. No. 4 probably gives a more accurate idea of the temperature. In all cases it is seen that towards the end of the reaction the temperature in the retort rises very high. In No. 3 the quick jumping of the temperature coincides with the violent oscillation of the manometer, each of which seem to be due to a nitre boil. Fig. 12 shows that in the case of trial No. 3 the temperature in the retort was lower than the temperature of the gases in the uptake until nearly at the end of the distillation.

Temperature of the waste gases from the fire.—Fig. 12 shows how these rise as the distillation proceeds, and in this case they follow fairly closely the rise of the temperature in the uptake. Fig. 13 gives a comparison of the waste gases in Trials 3, 4, and 5. It clearly shows that more heat is wasted in No. 4 with its high fire than in Nos. 3 and 5.

Conclusions.—The conclusion arrived at from the experiments is that in the first part of the distillation it is undoubtedly better to use a small fire. A good fire may be put on until the retort begins to make, and it is then to be turned low and not raised above a small fire until after the blow occurs. It has been shown that with a very small fire the retort makes acid rapidly at this period, and at the same time the blow, when it occurs, is a very mild one, which does not necessitate opening all the explosion doors with the consequent waste of heat. This fact of a good make of acid with small fire at the beginning is one of the most striking of the results of the experiments. If a large fire is put on, the distillation is quicker though not greatly so, as may be seen from Fig. 10. The extra heat seems to be mainly used in overheating the sides and top of the retort (and hence the gases coming from the retort) in heating the brickwork and in waste heat going out of the flue. The result of heating up the brickwork so much is that when the blow occurs the retort is still being heated strongly when the fire is put out, hence the blow is a violent one, and it is necessary to raise all the explosion doors to cool the retort. Even then a great deal of fume frequently blows out of the retort. The raising of the explosion doors is another cause for loss of heat. The advantage of faster distillation is far more than counter-balanced by the disadvantages and loss caused by the more violent blow and the greater quantity of gas used. Moreover, the retort requires close watching. If a large fire is on, the blow will begin and develop very quickly, and to prevent having very bad blows it is necessary to watch the retorts closely and consequently to employ more labour than would be necessary with the other mode of working.

After the blow is over the make of acid usually decreases rapidly, consequently it is necessary to watch the manometer and have the fire lit or raised, as the case may be, as soon as it indicates a negative pressure again.

From now onwards the best procedure to be followed is not so evident. By keeping the fire moderate until the end, the yield of strong acid is increased at the expense of the weak, but the time of distillation is increased by several hours. To maintain a constant make of acid all through, the fire would have to be raised very high towards the end, the amount of weak acid would be increased enormously, and after tapping the retort would be left very hot and it would require several hours' cooling before it could be charged again. Even then it is doubtful whether the make could be kept up to its first value. At the same time the temperature of the gases in the uptake would be very high, with consequent high temperature of the condensed acid and danger of cracking the peryware, also the decomposition of the acid would be high; above all the quantity of fuel used would be very great. Fig. 14 clearly indicates the difference in the fuel used in the two Trials 3 and 4, and gives an idea of the difference in driving fast at the end and going fairly slowly.

The most suitable method of working appears to be to raise the fire a little at a time at intervals of about 1½ hours, after the blow is over, but never to have more than a moderate fire on (1,500 cubic feet

per hour). The fire should be up to this height at about 9 hours after distillation commences, and kept there until ¼ of an hour before tapping, when it should be turned out. The retort should be tapped when the acid has a gravity of 1·310 at 15° C. It is strongly recommended that specific gravity of the acid should be taken after it has been cooled down and not when hot. Turning over to the weak main should take place when the gravity of the acid is at 1·450 at 15° C. The strength of the strong acid should then be 92-93 per cent., while the weak will be 60-65 per cent. The distillation should take about 15 hours. No. 3 took 16¾ hours, but 2¾ hours of this was spent in heating the retort up at first owing to its having been so cold to begin with. The time between lighting the fire and beginning to make should certainly not be more than 1 hour, and will nearly always be very much less except in cases where the retort has had to be left for some time between charges. Allowing one hour only for heating up, the time of No. 3 is reduced to 15 hours. Moreover the procedure recommended involves higher firing towards the end than was given to No. 3, so the time should not be more than the 15 hours. No. 3 had 0·9 per cent. HNO₃ left in the nitre cake, and this is high, but with the slightly increased firing recommended this should be reduced to 0·5 at most. Further than this it is not recommended that the distillation should be carried, as the weak acid obtained is not worth the increased time and fuel. The time necessary between distillation to allow the retort to cool down varies somewhat with the atmospheric temperature, and is a matter of experience. With the moderate fire at the end recommended here it will probably be possible to charge the retort as soon as the lid has been removed and the fume inside the retort escaped after tapping.

Particulars of trials.—The following are particulars of the five trials. The quantity of nitre used was in each case 1·50 tons. The quantity of sulphuric acid used was in each case such that the ratio $H_2SO_4 : NaNO_3$ was 1·05 : 1·00, or as near as could be estimated at the time.

The available HNO_3 was in each case calculated on the assumption that there was 99 per cent. $NaNO_3$ in the nitre.

Analysis of nitrate of soda (wet) :—

	Per cent.
H_2O	2·0
Insol.	0·09
Na_2SO_4	0·48
NaCl	0·81
$KClO_4$	0·04
$NaNO_3$	93·59
Moisture in dry nitre	0·3

Analysis of gas :—

	Per cent.
CO_2	5·7
CO	22·8
H_2	14·2
CH_4	4·5
N_2	52·8

B.Th.U. per cubic foot	-	-	-	$162 \cdot 5 = 90$ C.H.U.
Temperature of gas -	-		-	$45°$ C.
Analysis of waste gases :—		-		Per cent.
CO$_2$	-	-	-	- 7·9
O$_2$	-	-	-	- 10·6
Vacuum in waste gas flue	-	-	-	$0 \cdot 1$ inch of water

Trial No. 1.

Total time (fire lit until retort tapped)		-	13 hours 10 minutes	
Time heating up (fire lit until distillation commenced)	-	-	- 2 hours	
Mean temperature in uptake	-	-	$116°$ C.	
Strength of sulphuric acid used	-	-	89·0 per cent.	
Strong nitric acid	-	-	- 92·8 ,,	
			(0·6 per cent. HNO$_2$)	
Tons strong nitric acid -	-	-	1·070	
Tons HNO$_3$	-	-	- 0·993	
Percentage on available HNO$_3$	-	-	90·2 per cent.	
Weak nitric acid	-	-	- 58·3 ,,	
			(trace HNO$_2$)	
Tons weak nitric acid -	-	-	0·041	
Tons HNO$_3$	-	-	- 0·021	
Percentage on available HNO$_3$	-	-	1·9 per cent.	
Nitre cake H$_2$SO$_4$	-	-	- 35·6 ,,	
			(0·2 per cent. HNO$_3$)	
Tons nitre cake (calculated)	-	-	2·18	
Tons HNO$_3$ in cake	-	-	0·0044	
Percentage on available HNO$_3$	-	-	0·4 per cent.	

Nitrous acid in nitric acid :—

Tons HNO$_2$	-	-	-	0·0063 = 0·0084 tons as HNO$_3$
Percentage on available HNO$_3$	-	-	0·8 per cent.	
Total acid obtained (excluding HNO$_2$)	-	92·1 ,,	
Total acid obtained (including HNO$_2$)	-	92·9 ,,	
Total acid accounted for	-	-	93·3 ,,	

(HNO$_3$ and HNO$_2$ in strong and weak nitric acid and HNO$_3$ in nitre cake.)

Trial No. 2.

Total time (fire lighted until retort tapped)		11 hours 35 minutes	
Time heating up (fire lighted until distillation commenced) -	-	-	1 hour 25 minutes	
Gas used (estimated)	-	-	- 23,000 cubic feet	
Equivalent to cwts. of coal	-	-	4·6	
No. of times gas turned out	-	-	3	
Total time gas not burning -	-	-	1 hour 25 minutes	
Mean temperature of waste gases -	-	-	122° C.	
Strength of sulphuric acid used	-	-	87·9 per cent.	

Strong nitric acid - - - 92·8 per cent.
                                     (0·3 per cent. HNO_2)
   Tons strong nitric acid - - - 1·029
   Tons HNO_3 - - - - 0·955
Percentage on available HNO_3 - - 86·7 per cent.
Weak nitric acid - - - 58·3 ,,
                                     (0·2 per cent. HNO_2)
   Tons weak nitric acid - - 0·120
   Tons HNO_3 - - - 0·070
Percentage on available HNO_3 - - 6·4 per cent.
Nitre cake - - - - H_2SO_4 34·7 per cent.
                                   (0·1 per cent. HNO_3)
Tons nitre cake (calculated) - - 2·21
Tons HNO_3 in nitre cake - - 0·0022
Percentage on available HNO_3 - - 0·2 per cent.
Nitrous acid in nitric acid :—
   Tons HNO_2 - - - 0·0033 = 0·0044 tons
                                          as HNO_3
Percentage on available HNO_3 - - 0·4 per cent.
Total acid obtained (excluding HNO_2) - 93·1 ,,
Total acid obtained (including HNO_2) - 93·5 ,,
Total acid accounted for - - - 93·7 ,,

Trial No. 3.

Total time (fire lit until retort tapped) - 16 hours 45 minutes
Time heating up (fire lit until distillation
   commenced) - - - - 2 hours 45 minutes
Gas used (estimated) - - - 19,000 cubic feet
   Equivalent to, cwts. of coal - - 3·7
No. of times gas was turned out - - 2
Total time gas not burning - - - 56 minutes
Mean temperature of waste gases - - 107° C.
Mean temperature in uptake - - 109° C.
Strength of sulphuric acid used - - 87·8 per cent.
Strong nitric acid - - - 92·8 ,,
                                     (0·3 per cent. HNO_2)
   Tons strong nitric acid - - 1·0245
   Tons HNO_3 - - - 0·9504
Percentage on available HNO_3 - - 86·3 per cent.
Weak nitric acid - - - 60·3 ,,
                                     (0·1 per cent. HNO_2)
   Tons weak nitric acid - - 0·05 ,,
   Tons HNO_3 - - - 0·0320
Percentage on available HNO_3 - - 2·9 per cent.
Nitre cake - - - - H_2SO_4 35·6 per cent.
                                     (0·9 per cent. HNO_3)
   Tons nitre cake (calculated) - - 2·21
   Tons HNO_3 in nitre cake - - 0·0199

Percentage on available HNO_3 - 1·8 per cent.,
Nitrous acid in nitric acid :—
    Tons HNO_2 - - - 0·0032 = 0·0043 tons
                                         as HNO_f
Percentage on available HNO_3 - - 0·4 per cent.
Total acid obtained (excluding HNO_2) - 89·2 ,,
Total acid obtained (including HNO_2) - 80·6 ,,
Total acid accounted for - - - 91·4 ,,

Trial No. 4.

Total time (fire lighted retort until tapped)- 12 hours
Time heating up (fire lit until distillation
    commenced) - - - - 1 hour
Gas used (estimated) - - - 24,000 cubic feet
    Equivalent to cwts. of coal - - 4·7
No. of times gas was turned out - - 3
Total time gas not burning - - - 1 hour 6 minutes
Mean temperature in waste gases - - 126° C.
Mean temperature in uptake - - 124° C.
Strength of sulphuric acid used - - 91·2 per cent.
Strong nitric acid - - - 92·8 ,,
                          (0·5 per cent. HNO_2)
    Tons strong nitric acid - - - 0·9588
    Tons HNO_3 - - - - 0·8898

Percentage on available HNO_3 - - 80·8 per cent.
Weak nitric acid - - - - 63·2 ,,
                          (0·1 per cent. HNO_2)
Tons weak nitric acid - - - 0·1443
    Tons HNO_3 - - - - 0·0912
Percentage on available HNO_3 - - 8·3 per cent.
Nitre cake - - - - - H_2SO_4 32·9 per cent.
                          (0·7 per cent. HNO_3)
Tons nitre cake (calculated) - - 2·13
Tons HNO_3 in cake - - - 0·0149
Percentage on available HNO_3 - - 1·4 per cent.
Nitrous acid in nitric acid :—
    Tons HNO_2 - - - - 0·0048 = 0·0064 ton
                                 as HNO_3
Percentage on available HNO_3 - - 0·6 per cent.
Total acid obtained (excluding HNO_2) - 89·1 ,,
Total acid obtained (including HNO_2) - 91·1 ,,
Total acid accounted for - - - 91·1 ,,

Trial No. 5.

Total time (fire lit until retort tapped) - 14 hours 25 minutes
Time heating up (fire lit until distillation
    commenced) - - - - 1 hour 55 minutes
Gas used (estimated) - - - 23,000 cubic feet
    Equivalent to cwts. of coal - - 4·6

No. of times gas was turned out - .
Total time gas not burning -   -   - 5 minutes
Mean temperature in waste gases -   - 110° C.
Mean temperature in uptake -   - 103° C.
Strength of sulphuric acid used   -   - 91·4 per cent.
Strong nitric acid -   -   - 92·8   ,,
                               (0·6   ,, HNO)

  • Tons strong nitric acid -   -   - 1·025
  • Tons HNO_3 -   -   -   - 0·951

Percentage on available HNO_3 -   - 86·4 per cent.
Weak nitric acid -   -   - 70·0   ,,
                            (0·03 per cent. HNO_2)

  • Tons weak nitric acid -   -   - 0·064
  • Tons HNO_3 -   -   -   - 0·045

Percentage on available HNO_3 -   - 4·1 per cent
Nitre cake -   -   -   -   - H_2SO_4 33·0 per cent.
                            (1·1 per cent. HNO_3)

  • Tons nitre cake (calculated) -   - $2\frac{3}{4}$
  • Tons HNO_3 in cake   -   -   - 0·0235

Percentage on available HNO_3 -   - 2·1 per cent.
Nitrous acid in nitric acid :—

  • Tons HNO_2 -   -   -   - 0·0061 = 0·0082 ton
                                           as HNO

Percentage on available HNO_3 -   - 0·7 per cent.
Total acid obtained (excluding HNO_2) - 90·5 ,,
Total acid obtained (including HNO_2) - 91·2 ,,
Total acid accounted for   -   - 93·3 ,,

DESIDERATA IN RUNNING THE RETORT HOUSE

These may be conveniently summarised as follows :—

    1. *High production.*
    2. *Good recovery.*
    3. *Economy of fuel.*
    4. *A clean and well-kept plant.*

High production.—This will be secured by avoiding loss of time
in—

    (*a*) Charging nitre and sulphuric acid
    (*b*) Tapping and recharging.
    (*c*) Firing.
    (*d*) Distilling.

    Given the necessary labour the time between the end of one
distillation and the beginning of another should not exceed $1\frac{1}{2}$ hours.
    The draught on the plant should be very good, so that the retorts
can be recharged the moment tapping is finished. As soon as the
sulphuric acid is in, all dampers should be shut and a good fire started.

When the retort begins to make, the fire should be turned down very low or even extinguished.

Good recovery efficiency.—The following points must be, watched carefully :—

(a) Avoid leaks and blows.
(b) Do not distil at too high a temperature.
(c) Keep the absorption towers working properly.

Under this heading it should be noted that the coefficient of absorption for NO_2 falls off very rapidly above a strength of 60 per cent. It would be well therefore not to try to concentrate above 50 per cent. HNO_3 in the towers.

Economy of fuel.—Far too much fuel is frequently used. The temperature should *not* be regulated by opening dampers, but by cutting off the gas. A good fire should be used until the retort begins to distil. Immediately this occurs the fire should be cut down almost to a minimum and kept there *for 2 hours*. It can then be slightly raised for another hour, and after this gradually increased until after 9 hours distillation it is of a moderate size.

The fire should be extinguished 15 minutes before tapping.

Temperatures.

1st 4 hours to rise gradually from 95° C.–105° C.
2nd 4 hours to rise gradually from 105° C.–120° C.
3rd 4 hours to rise gradually from 120° C. 135° C.

The accompanying curves, Figs. 8–15, are the means of several experiments and will show the inter-dependence of rate of acid make, time, and temperatures.

Fig. 15 shows very clearly the point at which the retort should be switched on to the weak main. This point is 1·440 at 15° C. The temperature of the acid should be taken in several retorts and an average correction applied to all gravities. The finishing gravity should be 1·310 at 15° C., the temperature correction being 0·001 per 1° C.

These results will be secured by constant and unremitting attention to every detail, by watching very carefully the firing of the retorts, and by organising the labour so that the " dead time," *i.e.* the time between the end of one distillation and the beginning of the next distillation is reduced to the absolute minimum.

STANDARD RETORT CHARGES

Standard retort charges to obtain a nitre cake containing 30 per cent. of H_2SO_4 may be calculated from the following equations :—

$$NaNO_3 + H_2SO_4 = NaHSO_4 + HNO_3$$
$$85 \qquad 98 \qquad 120 \qquad 63$$
$$2NaNO_3 + H_2SO_4 = Na_2SO_4 + 2HNO_3$$
$$170 \qquad 98 \qquad 142 \qquad 126$$
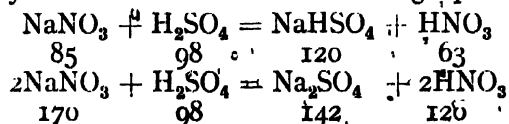
SPECIFIC GRAVITY OF ACID.

(MEAN OF 5 EXPTS)

Specific Gravity

Time in Hours
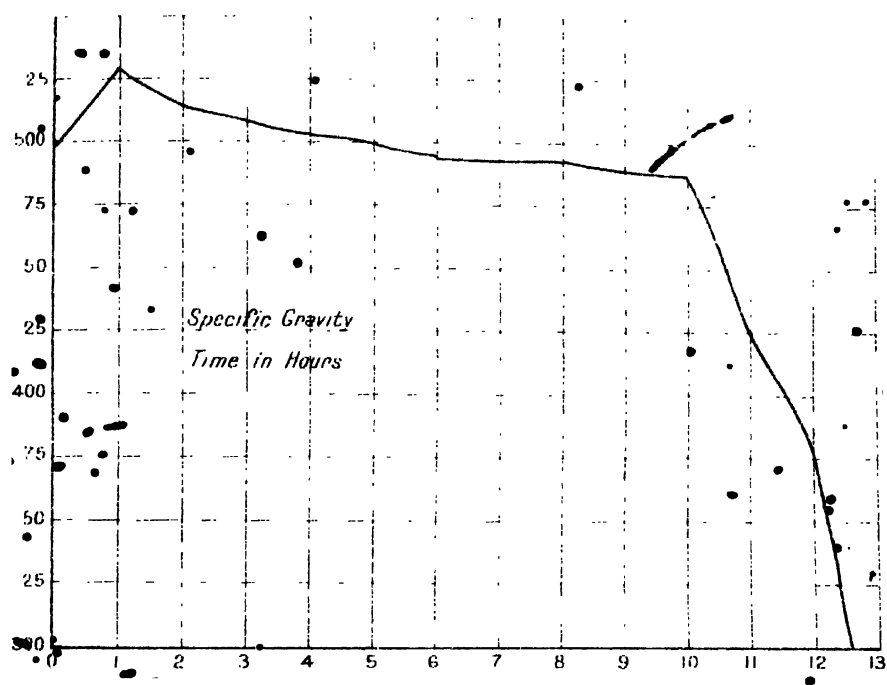
FIG. 15.

100 parts of nitre cake are equivalent to 30 parts of H_2SO_4, i.e. they contain $\dfrac{30 \times 120}{49} = 73 \cdot 5$ parts of $NaHSO_4$.

Hence, the composition required $=$ NaHSO$_4$ 73·5 per cent.
Na$_2$SO$_4$ 26·5

100·0

73·5 parts of NaHSO$_4$ are derived from —

$$\frac{73 \cdot 5}{120} \times 85 = 52 \text{ parts of } NaNO_3$$

and

$$\frac{73 \cdot 5}{120} \times 98 = 60 \text{ parts of } H_2SO_4$$

26·5 parts of Na$_2$SO$_4$ are derived from

$$\frac{26 \cdot 5}{142} \times 170 = 31 \cdot 7 \text{ parts of } NaNO_3$$

and

$$\frac{26 \cdot 5}{142} \times 98 = 18 \cdot 3 \text{ parts of } H_2SO_4$$

Hence, to obtain 100 parts of nitre cake of 30 per cent. acidity as H_2SO_4, the following quantities should be used, viz.:

Pure sodium nitrate (52 + 31·7) = 83·7 parts.
Pure sulphuric acid (60 + 18·3) = 78·3 parts.

For one ton pure $NaNO_3$, $\dfrac{78 \cdot 3}{83 \cdot 7} = 0 \cdot 935$ tons pure H_2SO_4.

The following table shows the amounts of sulphuric acid of various strengths required per ton of crude nitrate at varying degrees of purity which will yield a nitre cake containing 30 per cent. acid, reckoned as free H_2SO_4 :—

Purity of Crude Nitrate of Soda. Percentage NaNO$_3$.	Amounts of Sulphuric Acid required for 1 ton of Crude Nitrate. Quantities, in Tons.				
	100 per cent. H$_2$SO$_4$.	95 per cent. H$_2$SO$_4$.	94 per cent. H$_2$SO$_4$.	93 per cent. H$_2$SO$_4$.	92 per cent. H$_2$SO$_4$.
Per cent 98	0·916	0·964	0·975	0·985	0·996
97	0·907	0·955	0·965	0·975	0·986
96	0·898	0·946	0·956	0·966	0·976
95	0·888	0·935	0·945	0·955	0·965
94	0·879	0·925	0·935	0·945	0·955
93	0·870	0·916	0·926	0·936	0·946
92	0·860	0·905	0·915	0·925	0·935

DIAGRAM FOR USE IN CHARGING NITRIC ACID RETORTS

Fig. 16 shows a useful form of chart for use in charging nitric acid retorts, and is self-explanatory.

COMPOSITION OF SODIUM NITRATE USED

Representative samples of two consignments of sodium nitrate (Chili saltpetre) yielded the following analyses : –

			I.	II.
Sodium chloride	-	-	0·69	0·90
Sodium chlorate	-	-	0·02	0·03
Sodium sulphate	-	-	0·21	0·1c
Potassium chlorate		-	0·043	0·025
Calcium salts	-	-	0·00	0·00
Insoluble matter	-	-	0·16	0·04
Moisture	-	-	1·98	2·03
Sodium nitrate (by difference)		-	96·89	96·85

The variations in and mean values of the analyses of these two consignments covering a period of one month were- -

				I.	II.
Sodium chloride -	variations	-	-	0·39–1·10	0·67–1·17
	mean	-	-	0·81	0·84
Insoluble matter	variations	-	-	0·03–1·86	0·04–0·27
	mean	-	-	0·14	0·13
Moisture -	variations	-	-	1·46–4·63	2·03–2·8
	mean	-	-	2·82	2·0/

DETERMINATION OF CONDITIONS FOR MAXIMUM OUTPUT, CONDENSER EFFICIENCY, &C.

(Experiments carried out at H.M. Factory, Queen's Ferry, using Hart condensers).

A series of trials were carried out at Queen's Ferry to determine— ·
    (1) The maximum possible output.
    (2) Recovery efficiency of the condensing plant.
    (3) Optimum working conditions.

The arrangement of plant is shown in Fig. 17.

It is desirable that the receiver should have a capacity of not less than 40 gallons and have an outlet pipe of 1½ inches, otherwise there is choking when the plant is worked at its maximum output.

Arranged as shown, three cycles were carried out in one retort in 25 hours, that is, 3 × 2·25 = 6·75 tons of nitre were decomposed.

A Cambridge thread recorder was used in conjunction with two resistance pyrometers, one being inserted through the uptake and situated centrally in the pipe, whilst the other was placed in the vertical,

'EPORT ON HART RETORTS.

The object of this investigation was to determine:-

1. *Maximum possible output*
2. *Recovery efficiency of Condensing Plant*
3. *Optimum working conditions.*

→ Up-take

Silicon-iron Cascade →

ARRANGEMENT OF PLANT.

40 Gallon Measuring Vessel

Gauge Glass

40 Gallon Receiver

Acid Main

Hart Condenser

40 Gallon Measuring Vessel
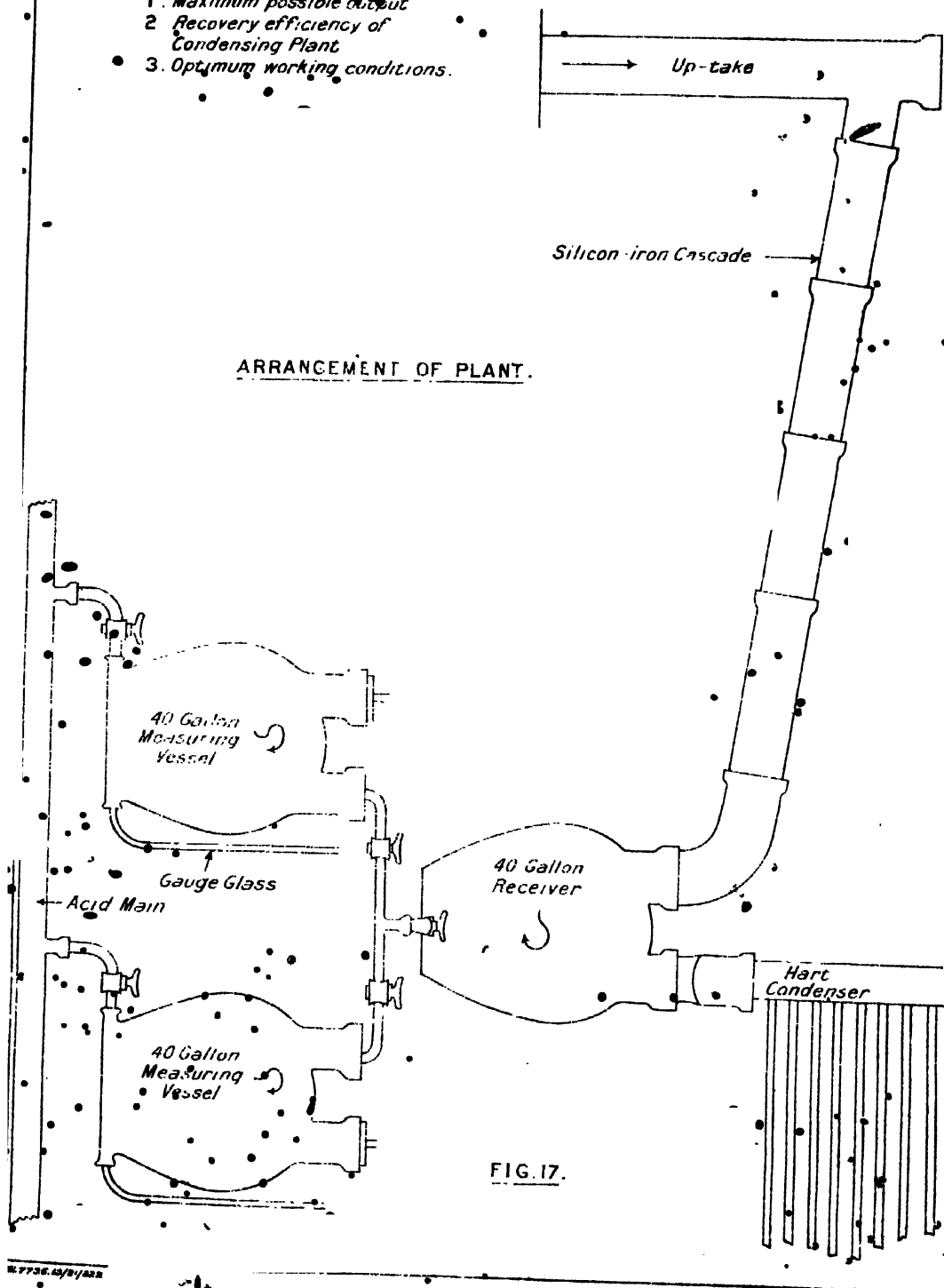
FIG. 17.

12 inches X 9 inches, waste gas flue at a point about 8 inches from the ground level.

Working conditions.—Working conditions were copied as closely as possible. Thus, in all cases except one, the experimental charge was started almost immediately after the preceding charge was tapped to ensure a warm retort.

In the case of charge 4, the retort was allowed to cool for about three hours before charging—the effect of this is discussed in the paragraph "Deductions from experiments," *vide infra*.

Measurement of raw materials.—The nitre in each case was weighed very carefully, and the weights can be guaranteed to within 7 lb. A sample of each charge was taken, and these mixed together and analysed. The result being :—$NaNO_3$, 98·00 per cent. by nitrometer on the dry salt; moisture, 1·5 per cent. This gives $NaNO_3$ in moist nitre as 96·5 per cent.; the figure 97·5 per cent. was used in calculating the sulphuric acid charges as the analysis was not carried out until the distillations were completed; the correct figure is used, however, for the estimation of nitric acid recovery.

The nitrometer method for determining the percentage of $NaNO_3$ is clearly the analogue of the process involved, and this figure should be taken as correct in preference to the refraction figure, which is always too high.

Optimum conditions of process.—Ideal running in a nitric acid plant requires :—

(1) Short time of cycle.

(2) Large percentage of strong acid made.

(3) High percentage of HNO_3 in this strong acid.

(4) Maximum recovery in condensers; the corollary being minimum recovery in absorption towers as weak acid.

(5) Dead time a minimum.

Short time of cycle.

It is always necessary to know the maximum possible output of any unit of plant, so that emergencies can be met by increased output.

With a view to determining this limit, charge 2 was started in a hot retort, and a full fire kept on throughout the run, the time occupied from commencement of charging to the finish of tapping was 7 hours 45 minutes; charge 3 was run in immediately afterwards, and had a cycle* of 7 hours 50 minutes; the retort was again charged and run together with the other units under the supervision of the shift chemist without individual attention, this charge occupied 9 hours 30 minutes. Thus three complete charges, each of 2·25 tons of nitre, were distilled and tapped in 25 hours.

The maximum output of a retort on the Hart system as installed at Queen's Ferry can be taken therefore as 4 tons of HNO_3 per day; ·

---

* The term cycle is taken throughout this report as the period elapsing from the commencement of charging to the finish of tapping.

this might be increased by running a larger charge than 2:25 tons of nitre.

Large percentage of strong acid.

On the assumption that the acid from the third pair of headers goes to the strong main, the best result was No. 4, which gave 96·5 per cent. strong acid expressed as HNO_3; Nos. 5 and 6 also gave high values, but in these cases the total recovery was low, which tends to negative the otherwise good results.

The meaning and causes of these results are discussed under the heading of " Deductions from experiments," *vide infra*.

High percentage HNO_3 in strong acid.

No. 4 was again the best with 92·2, but none gave results as high as might be expected, viz., 93–94 per cent. The low value of No. 5 is doubtless due to the low value of the sulphuric acid, which was caused by the sulphuric acid mixing plant not being able to supply 92·5 per cent. acid on account of the freezing of the 98 per cent. acid line, due to cold weather.

Dead time a minimum.

As a rule this is taken as meaning the time elapsing between the finish of one cycle and the commencement of the next; as the retorts are recharged immediately a charge is finished, the dead time is reduced to a few minutes per charge provided the supplies of nitre are forthcoming and the labour is fairly smart.

A more useful reckoning would be to count dead time as the sum of the time elapsing between the start of charging and the finish of tapping of the previous run, and the time actually occupied in charging and tapping. Table No. 1 shows that this time was executed regularly in 1 hour 15 minutes, which is fairly quick, the nitre charge being in position, and the cake pans empty as their turn came for use.

The removal of nitre cake is a serious factor; in the case of the three fast runs, the cake from the previous charge was not cool enough for removal when the next was ready to tap, so the cakes were run on top of the previous one, which had been liberally sanded to prevent sticking.

If the time of cycles is cut down much from their present value, it will be found necessary to surround the cake-pans with water to effect quicker cooling.

No. 6 charge was delayed 1 hour 10 minutes for tapping, in the other cases special arrangements for accommodation of cake were made.

Deductions from experiments. From examination of Fig. 18, which comprises the tabulated results of the various charges, it will be seen—

(1) That high speed of distillation does not necessarily mean low recovery;

(2) That strength of strong acid bears no relation to speed of distillation;

RECORDING PYROMETER CHARTS.

——— : Waste Gas Temperature
– – – : Uptake Temperature.

Charge Nº 5

Fire Lit

Distⁿ Began

Charge Nº 4.

Distⁿ Began

Fire Lit.

Time in Hours

Charge Nº 3.

Fire Lit

Distillation Began

Changing Began

Fire Out

Tapping Started

Charge Nº 2.

Fire Lit

Distillation Began

Changing Started

FIG. 19.

Malby & Sons, Lith.
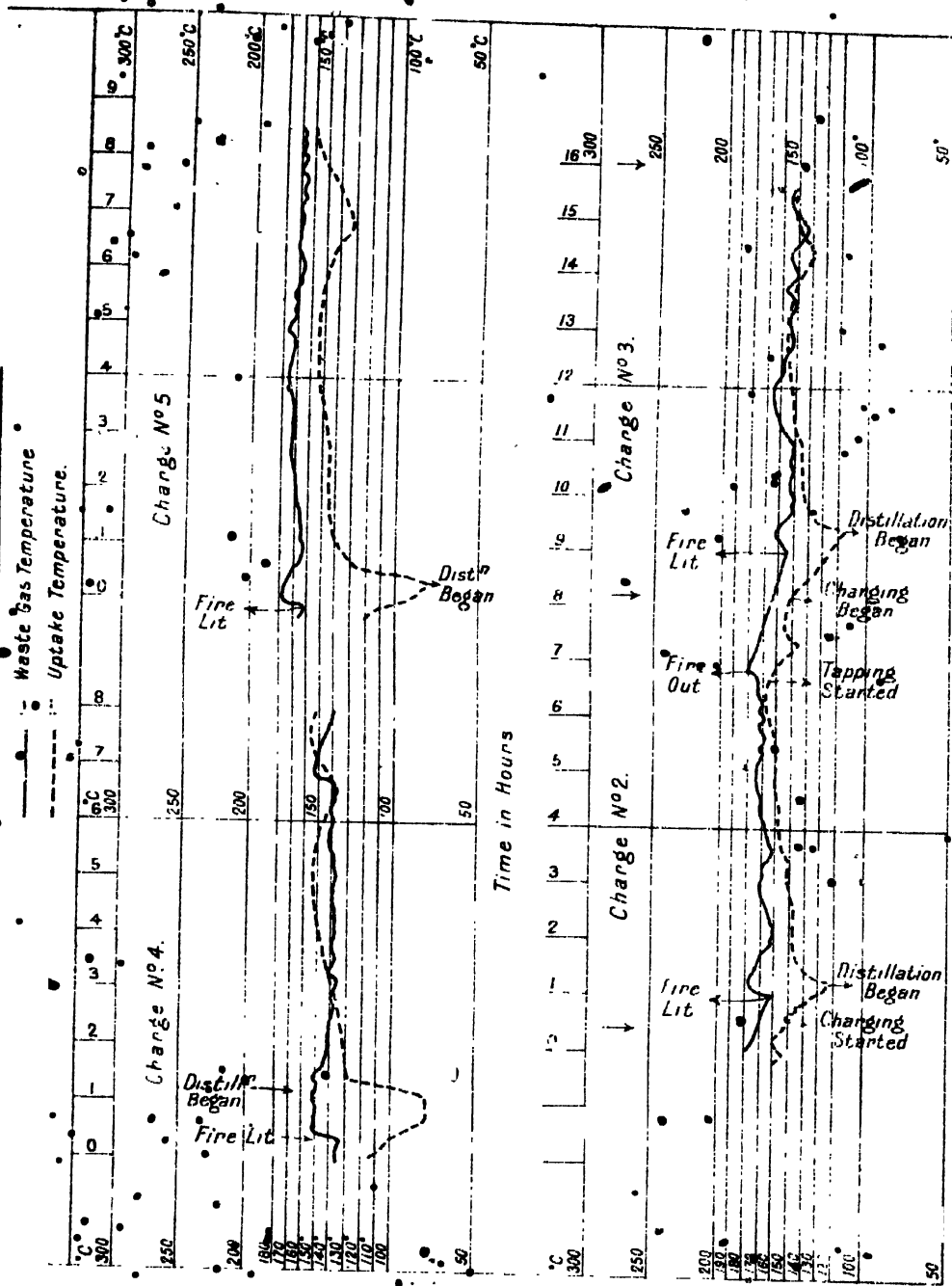
RECORDING PYROMETER CHART

CHARGE N° 6

------ uptake temp
——— waste gas temp.

200 190 180 170 160 150 140 130 120

FIG. 20.

RATE OF MAKE .... CHARGES N°? 2 AND 3.

N°2.
N°3.

1 Kilo - ·001 ton approx.

Lower curves represent make of third headers.

Rate of make in kilograms per minute.

Time of distillation in hours

FIG. 21.

RATE OF MAKE.

Shewn as kilograms/minute 1 Kilo = 00l tons approx.

Charge Nº 4 -------
" " Nº 5. ———

The shaded areas represent make of weak acid

The small curves at the bottom give make from 3rd headers.

Kilograms/minute

Weak

Hours of Distillation

FIG. 22

Malby & Sons.Lith
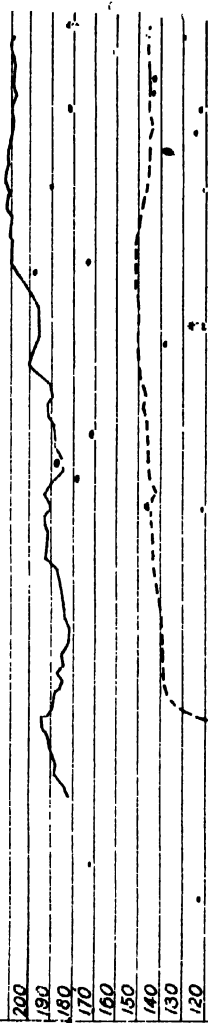
(3). That percentage recovery and strength of strong acid appear inter-dependent;

(4) That high temperature of waste gases from retort flue varies with (3) above.

This last important deduction is clear at once from the pyrometer charts, Fig. 19. In the case of charge No. 4 (the best from point of view of percentage recovery, percentage of strong, and percentage HNO_3 in strong) the flue gas temperature was below 140° C. during the greater part of the distillation, and was for a greater part of the time less than the up-take temperature. The reason for this is directly ascribed to the fact that the retort was allowed to cool for two or three days before the charge was started, in all other cases there was no such stoppage. The result of this being the cooling of the upper part of retort and surrounding brickwork.

Charge No. 5 was the worst for recovery, and percentage HNO_3, this latter is attributed to a slight extent to the weak sulphuric acid employed, but the flue temperature was 180° C. to 210° C., the highest value reached.

Charge No. 5 had a flue temperature of 170° C. to 180° C., and the next worst recovery.

These results point conclusively to the fact that if the top of the retort and surrounding brickwork are allowed to get above 140° C. as shown by waste gas temperature, then considerable decomposition of the acid vapours takes place.

The upper two flues do not contribute to the furtherance of distillation, but merely to keeping the upper part of the retort warm, preventing condensation of acid, and subsequent corrosion.

The methods that can be adopted to prevent this " cracking " of the acid vapours are as follows, viz., by altering the flue construction, so that the top of the two flues are in parallel to lower two, and by inserting a damper so that only sufficient hot gases can be admitted to keep the top of the retort at a reasonable temperat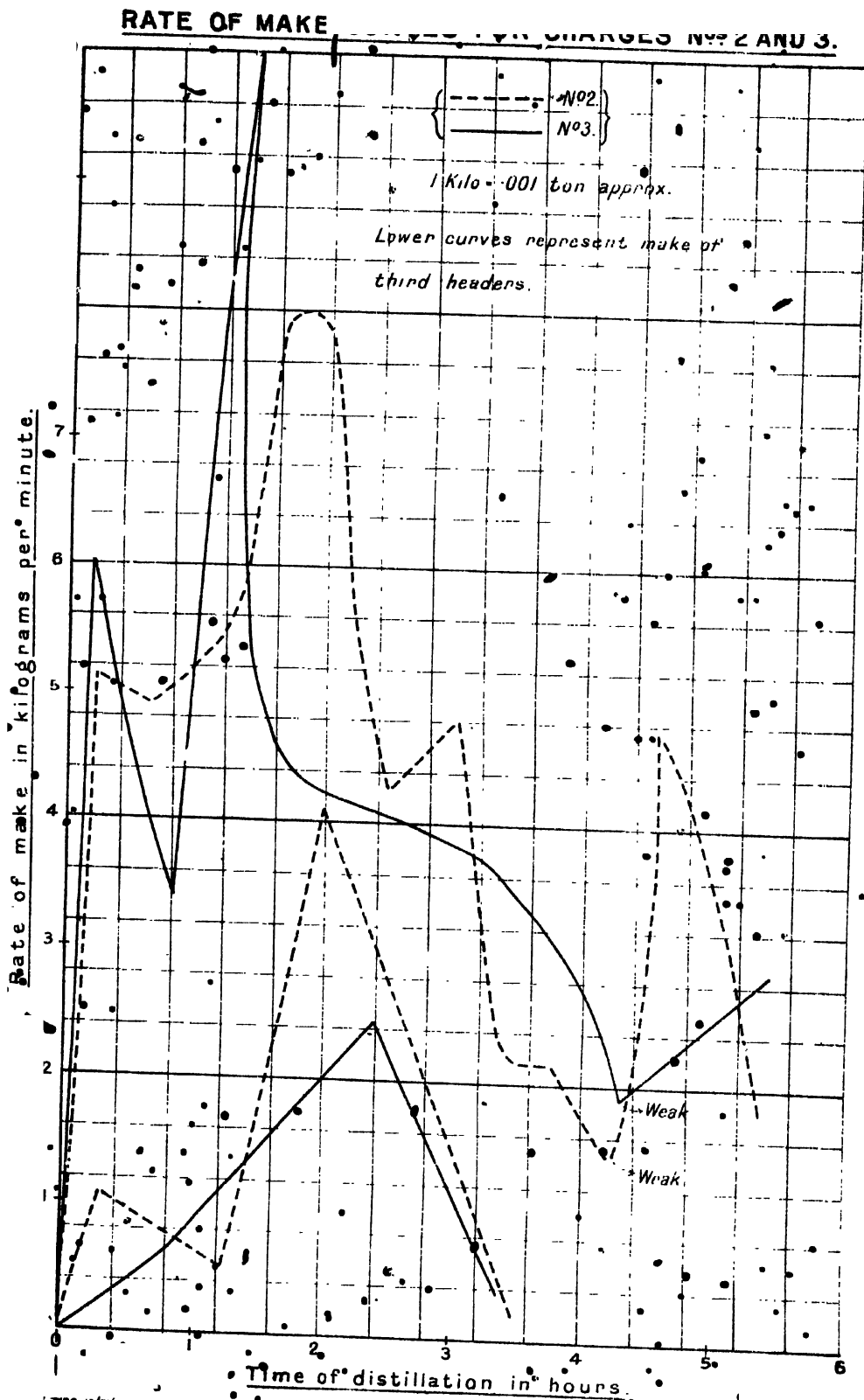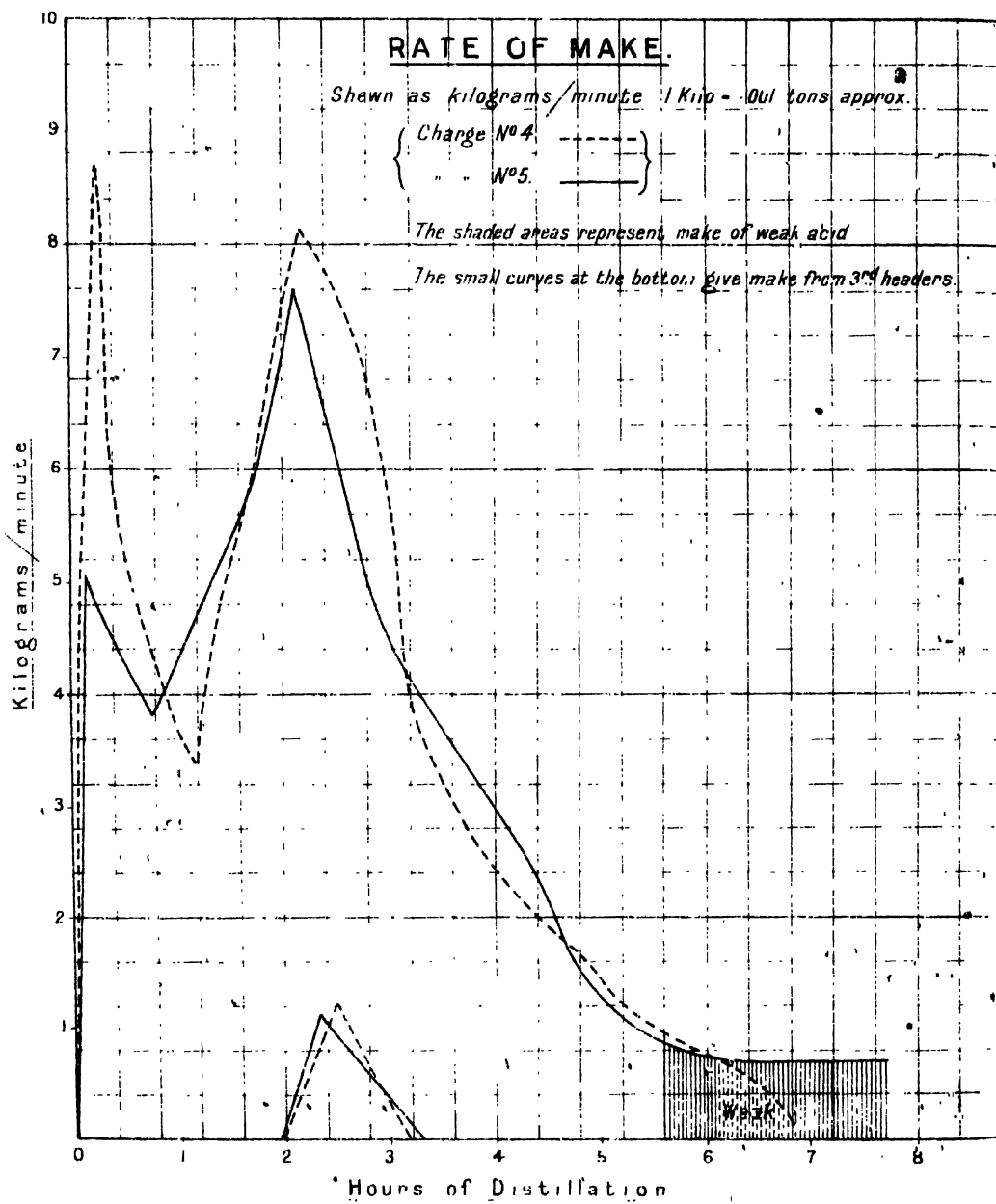ure, the main part of the heat passing through the lower two flues and consequently being concentrated on the lower part of the retort where it is required to assist distillation.

Gas and gas firing. The stack draught, measured on the downtake to the main flue, was fairly constant at the 0·15 inch of water, occasionally rising to 0·20-0·25 inch for shorter periods; this draught is sufficient to give good distillations provided that the gas pressure is maintained at 1 inch, as was the case during the experiments.

An average gas sample was taken during the running of charge 2 by means of an aspirator from the cleaning hole of the gas valve, at the rate of about 1 litre an hour, the result was—

		Per cent.
CO_2	- - - -	3·9
CO	- - - -	19·2
H_2	- - - -	8·2
CH_4	- - - -	4·9
N_2	- - - -	63·8

·Analysis of flue gases during the charge gave—

Hours of distillation.		Per cent. CO_2.	Per cent. O_2.
2 hours 0 minutes.	-	- 12·8	6·4
2 „ 30 „	-	- 10:0	9·6
2 „ 40 „	-	- 13·2	5·6
3 „ 0 „	-	- 14·0	4·8
4 „ 0 „	-	- 10·0	8·0
4 „ 30 „	-	- 13·0	3·6

The fires were constantly attended to, and an opening of $\frac{3}{4}$ inch for the air slide was found to give maximum efficiency. The above CO_2 figures amply demonstrate the good working of the fires.

Gas consumption.—An attempt was made in the case of No. 5 run to estimate the amount of gas consumed during the charge. The method employed was as follows :—

A continuous sample of the producer gas was taken by an aspirator from the cleaning hole of the gas valve, at about 1 litre an hour. At the same time a sample of the burnt gases was taken from the back flue at a spot just below the pyrometer, so that the temperature of the gases, passing through the sampling, could be ascertained.

These two samples were started as soon as the fire was lit, and stopped when it was turned out.

Both gas samples were carefully analysed, and these, together with the average value of the velocity of gases in the flue, taken by an anemometer, gave a value for the gas consumed.

Results: -

Average value of producer gas–

					Per cent.
CO_2	-	-	-	-	- 4·6
CO	-	-	-	-	- 16·9
H_2	-	-	-	-	- 8·0
CH_4	-	-	-	-	- 3·7
N_2	-	-	-	-	- 66·8

Average value of waste gases—

					Per cent.
CO_2	-	-	-	-	- 6·5
O_2	-	-	-	-	- 10·5

Average velocity of gases in flue, corrected to 15° C. = 5·51 ft./sec, the cross-sectional area of the flue being 108 square inches. Time during which fire was alight = 8 hours 45 minutes. 100 volume of producer gas of above composition yield, on combustion, 25·2 volume of CO_2.

Volume of waste gases passing through flue per sec.

$$= \frac{5 \cdot 51 \times 10^8}{144} = 4 \cdot 13 \text{ cubic feet per second.}$$

Since $6 \cdot 5$ per cent. of this is CO_2, and as the fire is alight during 31,500 seconds, the total volume of CO_2 formed is—

$$\frac{4 \cdot 13 \times 6 \cdot 5}{100} \times 31,500 = 8,456 \text{ cubic feet.}$$

Since $25 \cdot 2$ cubic feet of CO_2 are formed from 100 cubic feet of producer gas,

$$\text{Producer gas consumed} = \frac{8,456 \times 100}{25 \cdot 2} = 33,560 \text{ cubic feet.}$$

The usual figure taken for the volume of producer gas formed from a ton of coal is 140,000 150,000 cubic feet at N.T.P., but as the gas is low in carbon monoxide, the higher figure will be taken.

On this assumption, coal used $= 0 \cdot 224$ ton, and since the HNO_3 produced was $1 \cdot 331$ tons, then coal used per ton HNO_3 produced $= 0 \cdot 168$ ton.

The average figure for tons of coal per ton HNO_3 for period 17th November 1916 to 17th December 1916 is $0 \cdot 247$ ton for the Hart retorts. Allowing even considerable margin for experimental errors, it is quite evident that the fuel consumption can be considerably cut down.

Saving in gas can be effected by greater attention to firing. Under existing conditions the fires do not receive the share of attention they deserve as the prime movers of the whole process.

The proper manipulation of gas firing requires skilled labour. When these tests were carried out the care of the fires was almost wholly in the hands of girls, who were quite unsuited for the work, except for such things as cleaning out the valves.

Nitre cake.—As seen from table, Fig. 18, the acidities of the nitre cakes were rather irregular despite the greatest precautions with measuring the nitre and sulphuric acid.

In each case the sulphuric acid was sampled and analysed; the remaining factor, which is variable, being the percentage of moisture in the nitre.

The charges of sulphuric acid were calculated on a $97 \cdot 5$ per cent. $NaNO_3$ basis, and in every case except No. 3 this proved too high.

As was pointed out in the section on raw materials, the average analysis of the nitre charges was $98 \cdot 00$ per cent. of $NaNO_3$ on the dry salt by the nitrometer, and $1 \cdot 5$ per cent. of moisture. This corresponds to $96 \cdot 53$ per cent. of $NaNO_3$ on the moist nitre.

It is quite impossible to get cakes regularly low in H_2SO_4 until the nitre and sulphuric acid are both standardised. If the drier is worked efficiently, the moisture variation can be eliminated, and the remaining factor is the sulphuric acid, with regard to which the chemist in charge

must know the strength of acid he is being supplied with at the moment. If large mixes of feed acid were made at a time, and this properly sampled and analysed, there is no reason why cakes containing 29–30 per cent. H_2SO_4 should not be the rule rather than the exception.

Nitric acid in nitre cake.—The presence of nitric acid in the cake is undesirable from the point of view of the nitric acid manufacturer, as it is a cause of loss of acid, and also from the point of view of the nitre cake consumer, since it acts as a troublesome impurity.

When charge No. 1 was run, it was the practice on the plant to tap at a gravity of distillate of 1·340, not taking the temperature into account. Charge No. 1 was tapped at 1·340, the temperature of the acid being 70° C., and the cake was found to contain 1·15 per cent. HNO_3. Subsequent charges were tapped at lower gravity, but all contained more or less nitric, the lowest being No. 2 with 0·15 per cent. HNO_3, which was tapped at 1·279, the temperature in this case being 87° C.

If cakes free from nitric are required, it is necessary to make a temperature correction, or to specify a specific gravity corresponding to the maximum temperature likely to be reached, the former method would be most economical from the point of view of saving time.

It would seem that to tap at 1·250 regardless of temperature would give good results, but far better would it be to tap at 1·350 at 15° C., allowing a correction of 0·008 for every 5° C.

In the same way, the point at which the retort is changed to weak should also be corrected for temperature, 1·470 at 15° C. has been found to give good results.

The functions of the 3rd pair of headers.—The figures show that the 3rd headers deliver acid only at the period of greatest make of the first two headers, and then it is an acid of average strength 92·7 per cent. HNO_3. It is to be expected that these headers would give strong acid, since weaker acid, having the highest boiling point, would condense in the first two banks of tubes, and the stronger acid of boiling point 80°–90° C., would pass on to the third headers. It is clear that these headers should be connected to the strong main instead of to the weak.

The very high percentage of nitrous in this acid is most marked, and is due to the low temperature prevailing in the third header. It was found that the temperature at the exit to the fume main was usually about 10° C., occasionally falling to 5° C. when the cooling water was particularly cold; at the time of maximum make this temperature would rise to 20°–25° C. These low temperatures are quite sufficient to condense large amounts of N_2O_4. The only way to reduce this would be not to cool the third header at all, which would doubtless lead to slightly higher losses to the absorption towers, but would seem the only method available where nitrous is an objection.

Acidity of gases to fume main.—The percentage of gases soluble in caustic soda, in the exit to the fume main from the third header, was determined by the Orsat apparatus, also the amount of free oxygen present.

.The determinations were made just after a slight blow during the running of charge No. 5, the steady fall of the acidity shows the cessation of the blow :—

Time.		Percentage of gases soluble in NaOH.	Percentage of oxygen.
During blow.	- -	43·4	11·6
10 minutes later	- -	26·4	16·2
20 ,, ,,	- -	17·2	17·2
30 ,, ,,	- -	9·0	18·4
40 ,, ,,	- -	6·0	20·2
1 hour 25 minutes	- -	5·4	19·8

The source of the free oxygen is a matter of surmise, considering the poor draught it is hardly likely to be air which is sucked in through joints, so presumably it is due to decomposition of NO_2.

The draught on the flue was not sufficient to get an anemometer reading. If an investigation would have been made in this direction with a view to getting a value for the losses to the absorption towers.

Summary of conclusions and further trial run. —These experiments indicate very clearly that the condensing system is very good, and that the gas firing and general arrangements of the flues are efficient. Thus three complete cycles are performed in 25 hours.

Further improvement in this direction seems neither advisable nor possible.

It is not possible because the nitre cake cannot be removed sufficiently fast, and it does not seem advisable because destructive distillation undoubtedly occurs when retorts are forced beyond a certain speed.

Further, the wear and tear on the plant must be considered. The first desideratum is that the plant should produce the required tonnage of HNO_3.

The maximum quantity required is taken as 2·8 tons HNO_3 per retort per day.

This tonnage would be produced by a cycle for 2¼ tons of nitre of 14 hours, so that in working for a 12 hour cycle should be well within the requirements.

The 12 hour cycle could be divided into 8 hour for distillation and 4 hour for other operations—a fair allowance if more rapid methods of removing nitre cake from the nitric acid factory are adopted.

Analysis of the results shown in the table indicate clearly that the loss of HNO_3 is too high.

Since this loss is caused by decomposition of HNO_3 in accordance with the following equation—

$$4HNO_3 = 2H_2O + 2NO_2 + O_2$$

The acid condensed in the Hart condenser must be weaker than it would otherwise be.

Thus we have—

(1) Actual loss of HNO_3.

(2) Additional consumption of oleum and subsequent additional sulphuric concentration consequent on the formation of water in the retort by decomposition of HNO_3. This decomposition can be caused by (1) too slow a distillation; and (2) too rapid a distillation.

In between is a set of optimum conditions which can be regarded as being embodied in, and guaranteed by the results of the large scale experiment detailed below.

Six retorts on a separate main were taken and put into a reasonable order.

These were charged in rotation at intervals of two hours. The acid made was all run into a pair of lead receivers. At the end of the run this acid was carefully agitated by means of an air jet and was carefully sampled and measured.

Since these lead tanks had been accurately calibrated, the figures given can be taken as representative and reliable.

Method of firing.—Immediately after charging, each retort was fired heavily, and as soon as distillation commenced the fire was cut down to a quarter fire and the distillation allowed to proceed for four hours under its own heat.

At the end of four hours the fire was gradually increased until distillation was finished.

Retort No.	Charging commenced.	Charging finished.	Distillation commenced.	Distillation stopped.	Time of distillation.	Sp. gr. of acid when distillation stopped.
	p.m.	p.m.	p.m.	a.m.	h. m.	
5	3.30	4.10	4.55	12.15	7 20	1·310
3	4.55	5.35	6.10	3.15	9 5	1·310
4	6.45	7.20	7.45	2.30	6 45	1·310
2	8.30	9.10	10.25	7.30	9 5	1·280
6	9.30	10.5	10.55	7.40	8 45	1·310
1	10.5	10.40	11.55	8.20	8 25	1·310

Average time of distillation = 8 hours.

Make of acid.—Lead receivers Nos. 1 and 2 were emptied as far as possible for this test. They contained the following HNO_3 before starting :—

No. 1 :—1¾ inches at 84·2 per cent. HNO_3
and 1·475 sp. gr. -   -   -   -   0·27 ton HNO_3

No. 2 :—1½ inches at 81·1 per cent. HNO_3
and 1·465 sp. gr. -   -   -   -   0·22 ton HNO_3

Total   -   -   -   -   0·49 ton HNO_3

No. 1 receiver was used first and was filled to 30½ inches by 2.20 a.m., when the run was turned into No. 2.

No. 1 receiver was agitated with a single air-jet for 1¼ Hours, when samples were taken by means of glass dipping tubes; No. 2 receiver was treated similarly.

No. 1 receiver.

Laboratory analysis :

$$\left.\begin{array}{l} \text{HNO}_3, \ 90\cdot \text{ per cent.} \\ \text{HNO}_2, \ \text{? per cent.} \\ \text{Sp. gr. at } 15^\circ\text{C. } 1\cdot492 \end{array}\right\} 30\tfrac{1}{2} \text{ inches at} \left\{\begin{array}{l} 90\cdot0 \text{ per cent. HNO}_3 \text{ and } 1\cdot492 \text{ sp. gr.} \\ 5\cdot61 \text{ tons acid } 1\cdot492 \text{ sp. gr.} \\ 5\cdot05 \text{ tons HNO}_3 \end{array}\right.$$

No. 2 receiver.

Laboratory analysis : —

$$\left.\begin{array}{l} \text{HNO}_3 \ 83\cdot3 \text{ per cent.} \\ \text{HNO}_2 \ \text{trace per cent.} \\ \text{Sp. gr. at } 15^\circ \text{ C. } 1\cdot473 \end{array}\right\} 23\tfrac{1}{4} \text{ inches at} \left\{\begin{array}{l} 83\cdot3 \text{ per cent. HNO}_3 \text{ at } 1\cdot473 \text{ sp. gr.} \\ 4\cdot27 \text{ tons acid at } 1\cdot473 \text{ sp. gr.} \\ 3\cdot55 \text{ tons HNO}_3 \end{array}\right.$$

Deduct quantity in receivers originally, which gives 8·11 tons HNO₃ as produced.

Quantities charged = 13·5 tons of nitre at 97 per cent. NaNO₃ = 13·1 tons NaNO₃, which are equivalent to $13\cdot1 \times 0\cdot741 = 9\cdot707$ of HNO₃.

Quantities produced and condensed.

From 1st headers - - - - 8·110 ton,
From 2rd headers - - - - 0·276 ton,
                                    8·386 ton,

.e., 86·6 per cent. recovered by condensation.

A recovery of 86·6 per cent. by actual condensation is reasonably good, and if to this be added 5·5 per cent. estimated recovery from the absorption towers, the total yield in these experiments is not less than 92·1 per cent.—a good figure which may be regarded as both desirable and obtainable.

Nitre cake acidities. —The irregular feeding of the Cummer drier gives a dry nitre of varying. moisture content which would not be detected by a daily sample.

This variation has 2 per cent. limits— sufficient, in fact, to upset such a careful adjustment as is necessary for controlling the nitre cake acidity.

If roller feeds are installed this variant should disappear.

A consideration of desired economy in nitric acid manufacture at Queen's Ferry.

Three Hart retort houses = 150 tons HNO₃ daily.

For fortnight ending December 19th, 1916, loss of HNO₃ at Hart house was 13 per cent. and the coal consumption o 24 ton per ton of HNO₃.

Average nitre cake acidity = 32·5 per cent. H₂SO₄.

Nitric acid losses.

Assume a 6 per cent. loss of HNO$_3$ as representing fair working, we have an additional loss of 7 per cent.

On the total ultimate HNO$_3$ output of the factory this would be 10·5 tons daily.

∴ HNO$_3$ losses = 10·5 tons per day.

Fuel losses.

The figure 0·24 ton per ton HNO$_3$ is too high—0·15 should be obtainable.

∴ avoidable loss = 0·9 ton of coal per ton of HNO$_3$, or 13·5 tons per day.

Sulphuric acid loss in nitre cake.

Assuming 29 per cent. as a reasonable figure.

Our avoidable loss = 32·5 — 29 = 3·5 per cent. H$_2$SO$_4$ on nitre cake.

Present approximate daily weight of nitre cake = 260 tons.

∴ loss of H$_2$SO$_4$ = $\frac{260}{100}$ × 3·5 = 9·1 tons of H$_2$SO$_4$.

Total *avoidable* losses per day on the ultimate output of the three retort houses and based on past working of the Hart retort house.

By HNO$_3$ losses   -   -   -   -   10·5 tons per day.
By fuel losses   -   -   -   -   13·5 tons per day.
By nitre cake (H$_2$SO$_4$)   -   -   -   9·1 tons per day.

The value of the attached investigation and the necessity for the immediate application of the principles enumerated is strikingly shown by these figures.

FURTHER REPORT ON NITRIC ACID PLANT WITH HART CONDENSERS

Efficiency of condensing system.—This was determined by –

(1) Actual measurements of acid recovered from absorption towers.

(2) Physical and chemical determination of the velocity and composition of the gases entering and leaving the towers.

It will be seen that concordant results were obtained, which pointed to efficient working.

Velocity of gases in 9 inch main entering towers = 6·92 feet per second.

Velocity of gases entering ejector (*i.e.* leaving last tower) = 17·1 feet per second.

Gases entering towers $\begin{cases} NO_2 = 3\cdot4 \text{ per cent.} \\ N_2O = nil. \\ O_2 = 19\cdot4 \text{ per cent.} \end{cases}$

Gases leaving towers

$$-\left\{\begin{array}{l}NO_2 = 0\cdot4 \text{ per cent.}\\ NO = nil.\\ O_2 = 20\cdot6 \text{ per cent.}\end{array}\right.$$

$$4NO_2 + O_2 + 2H_2O = 4HNO_3$$

i.e. 22·2 litres give 63 gm. HNO_3.

1 litre gives $\dfrac{63}{22\cdot4}$ gm.

cubic foot gives $\dfrac{63 \times 28\cdot3}{22\cdot4 \times 454} = 0\cdot175$ lb.

Volume of gas *entering* towers per second through the 9 inch pipe

= Cross-section × velocity.

= 0·442 × 6·92 = cubic feet per second.

= 0·442 × 6·92 × 60 × 60 × 24 = 264,200 cubic feet per day.

Of this, 3·4 per cent. is NO_2, *i.e.* $\dfrac{264200 \times 3\cdot4}{100} = 8,990$ cubic feet per day.

∴ equivalent weight of HNO_3 entering tower per day

$$= \dfrac{8990 \times 0\cdot175}{2240} = 0\cdot710 \text{ ton } HNO_3$$

Similarly, volume of gases *leaving* towers

$$\dfrac{0\cdot422 \times 17\cdot1 \times 60 \times 60 \times 24 \times 0\cdot4 \times 0\cdot175}{100 \times 2240} = 0\cdot294 \text{ ton } HNO_3$$

HNO_3 formed in absorption towers = 0·504 ton HNO_3

Average daily production = 48 tons HNO_3

HNO_3 recovered in towers $= \dfrac{0\cdot504 \times 100}{48} = 1\cdot05$ per cent.

HNO_3 lost through exit $= \dfrac{0\cdot204 \times 100}{48} = 0\cdot43$ per cent.

Actual measurements over a period of two days gave :—

1st day.—2·18 tons at 76·8 per cent. = 1·7 tons HNO_3

2nd day.—2·4 tons at 82·6 per cent.   2·0 tons HNO_3

During this period acid produced was 96 tons HNO_3

∴ Acid from towers = 3·8 per cent. of total.

This includes the acid condensed in the 3rd header, which by measurement was found to be approximately 2 per cent of the total make.

∴ Acid made in absorption towers = 1·8 per cent. of total.

It is evident therefore that on the two days in question the retort house was being run quite efficiently. As a matter of fact the recovery for that week was 95 per cent. of theory.

The remaining 3·2 per cent. loss can be partly accounted for by leaks and by formation of unrecoverable nitrosyl chloride ($NOCl$).

Fuel economy and the life of the plant.—In the preceding report the pyrometer determination of the waste flue gases and of the nitric acid vapours pointed to superheating of the latter.

A series of temperatures on several retorts were taken therefore, and the results are shown on the attached graphs (Figs. 23–28).

They show clearly that superheating does occur.

Conditions.—The whole of the retorts were run as usual by a shift of girls and men, in charge of a shift chemist, all of whom had considerable experience with the plant.

In no case, except in one instance especially noted later, was anything altered from usual working conditions; the data being obtained from several retorts over a period of days and at the convenience of the observer.

Experience on uptake and cascade pipes.—The observed temperatures on the uptake pipes appeared to be rather high, so several samples of the gas were examined at this point for nitric oxide (NO).

It was found to be a rather difficult matter to obtain anything like a constant volume of gas in an Orsat apparatus owing to condensation of nitric acid in the capillary tubes, &c., so no figures are given.

In no case was any trace of nitric oxide or oxygen detected. Manometer tubes were inserted before and after every bend in the cascade pipes and carefully observed.

No difference in the manometer readings could be detected, so that apparently the bends on the cascade pipes are sufficiently wide to prevent gas chokes.

An earlier report contained a suggestion to by-pass the waste gases in order to prevent superheating of the top of the retort.

Superheating of the top of the retort can be partly controlled by reducing the speed of the gases in the waste flue.

Curves are appended (Figs. 23–28) showing the relation between the " age " of the retort (time taken when the charge, complete with the sulphuric acid, is in and the fires are lit) and the waste flue temperature.

On the same sheet is plotted a curve showing the relation between the age of the retort and the temperature of the acid vapours issuing therefrom.

The whole of the curves show one complete cycle of the retort. Fig. 27 appears to yield the best result. It is a retort already mentioned, the furnace of which was especially attended to by the chemist in charge of the house and a carbon dioxide value of 10 per cent. obtained in the waste gases from flue.

It is seen that the temperature of the uptake never exceeded 153° C. after distillation had commenced, the higher temperature recorded for the first two hours being due to the previous charge. Similarly the highest temperature recorded in the waste flue was 215° C. when distillation was complete. The time of cycle was 11¼ hours.

Fig. 28 records the shortest cycle, the time being in this case 8½ hours. The temperature of the gases leaving the retort oscillates a good deal,

Age of Retort- Hours after acid in

FIG. 23.

Age of Retort - Hours after acid in.

FIG. 24.

— Waste flue temperature
- - - Uptake temperature

Waste flue temperature
Uptake temperature

Age of Retort — Hours after acid in.

FIG. 25.

Waste flue temperatures
Uptake temperatures

Age of Retort, Hours after acid in

FIG. 26.

Waste flue temperatures.
Uptake temperatures.

Age of Retort – Hours after acid in

FIG. 27.

Age of Retort - Hours after acid in.
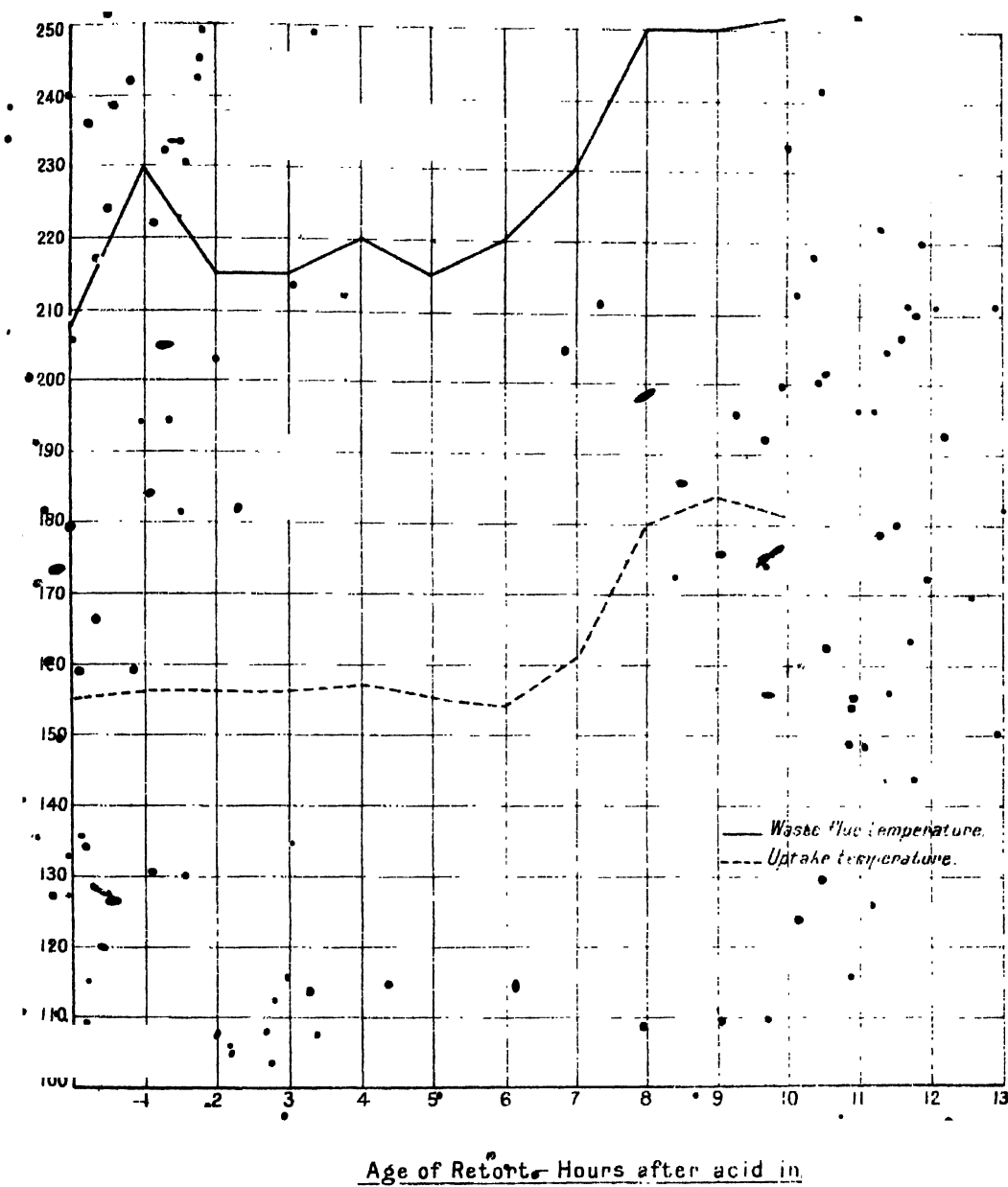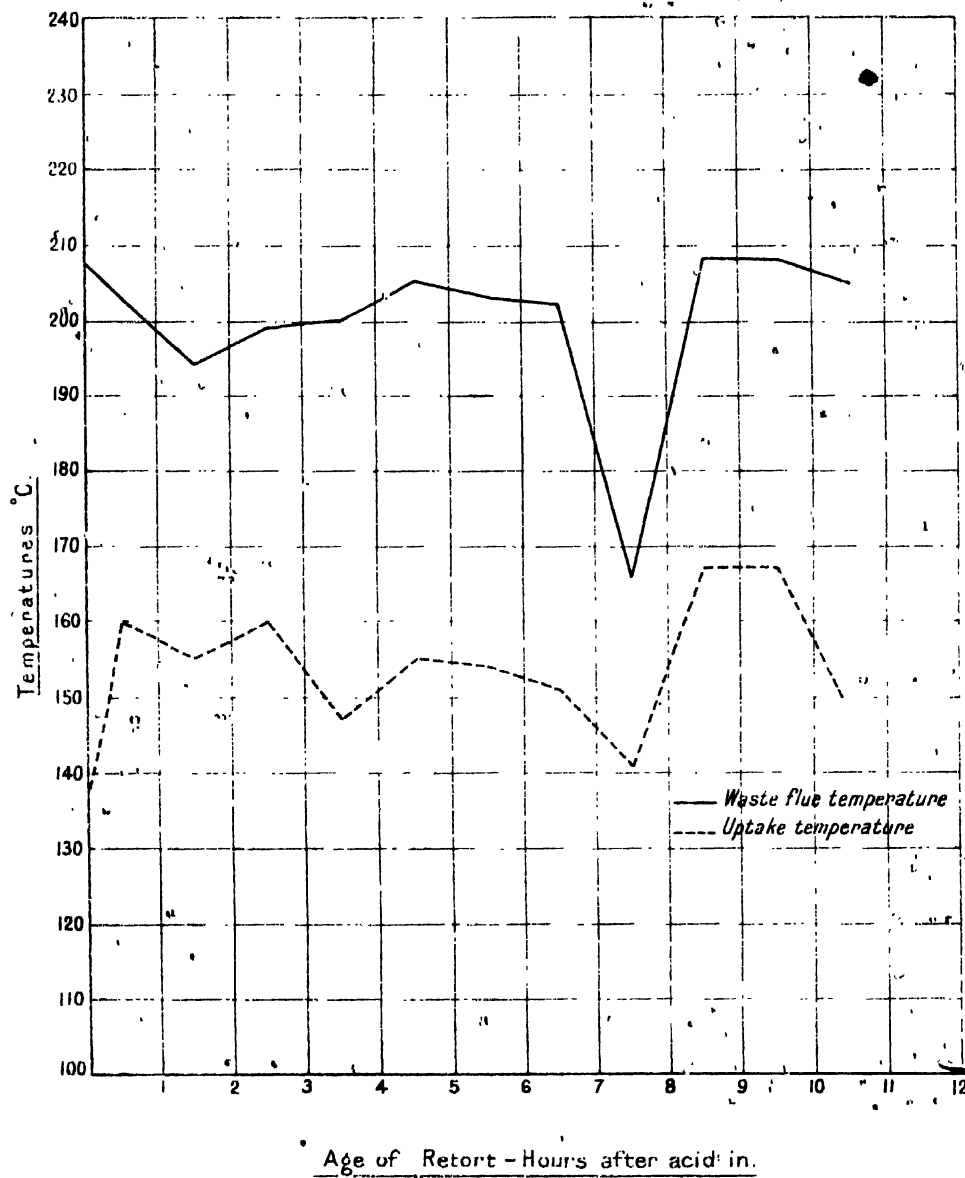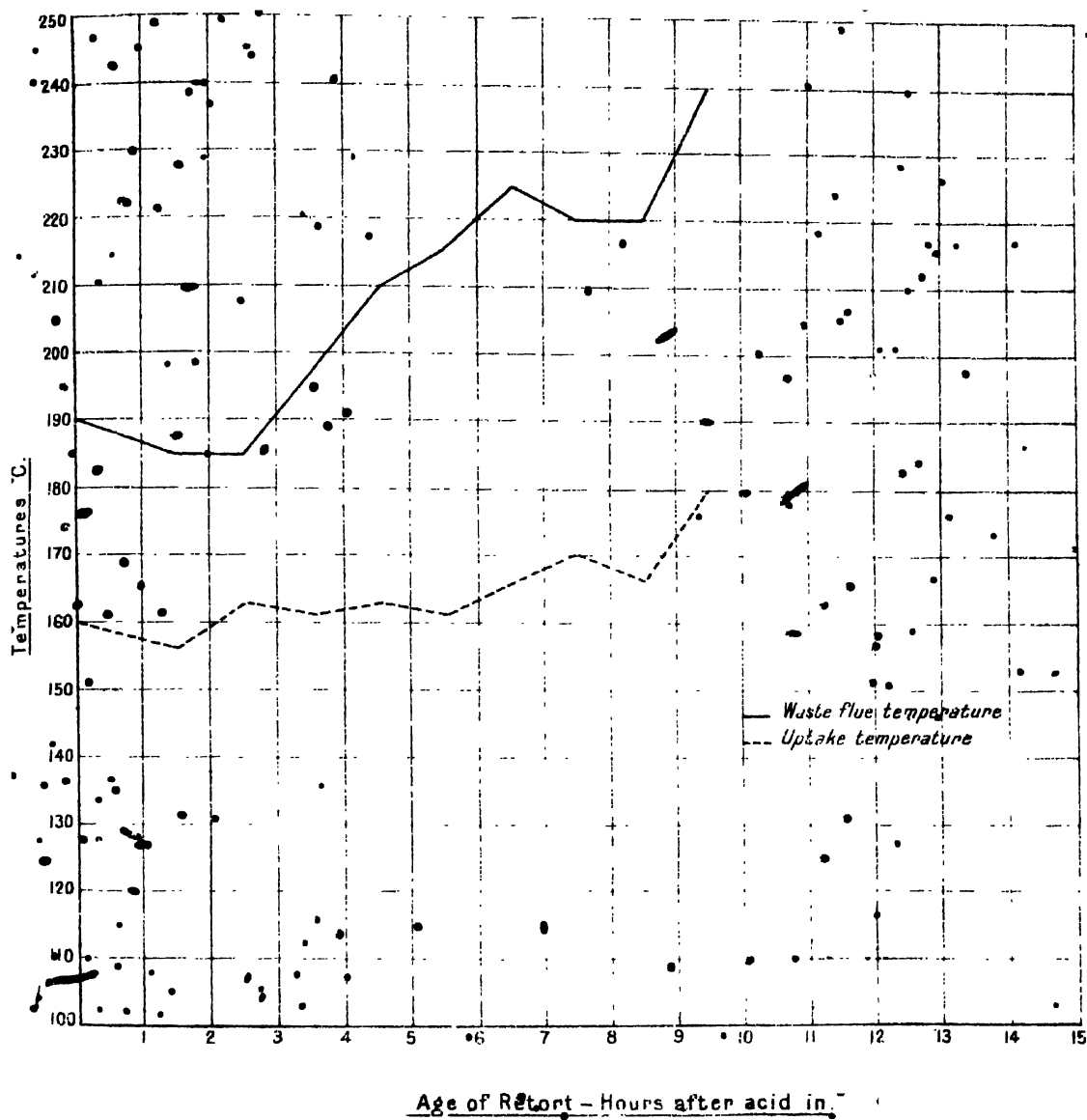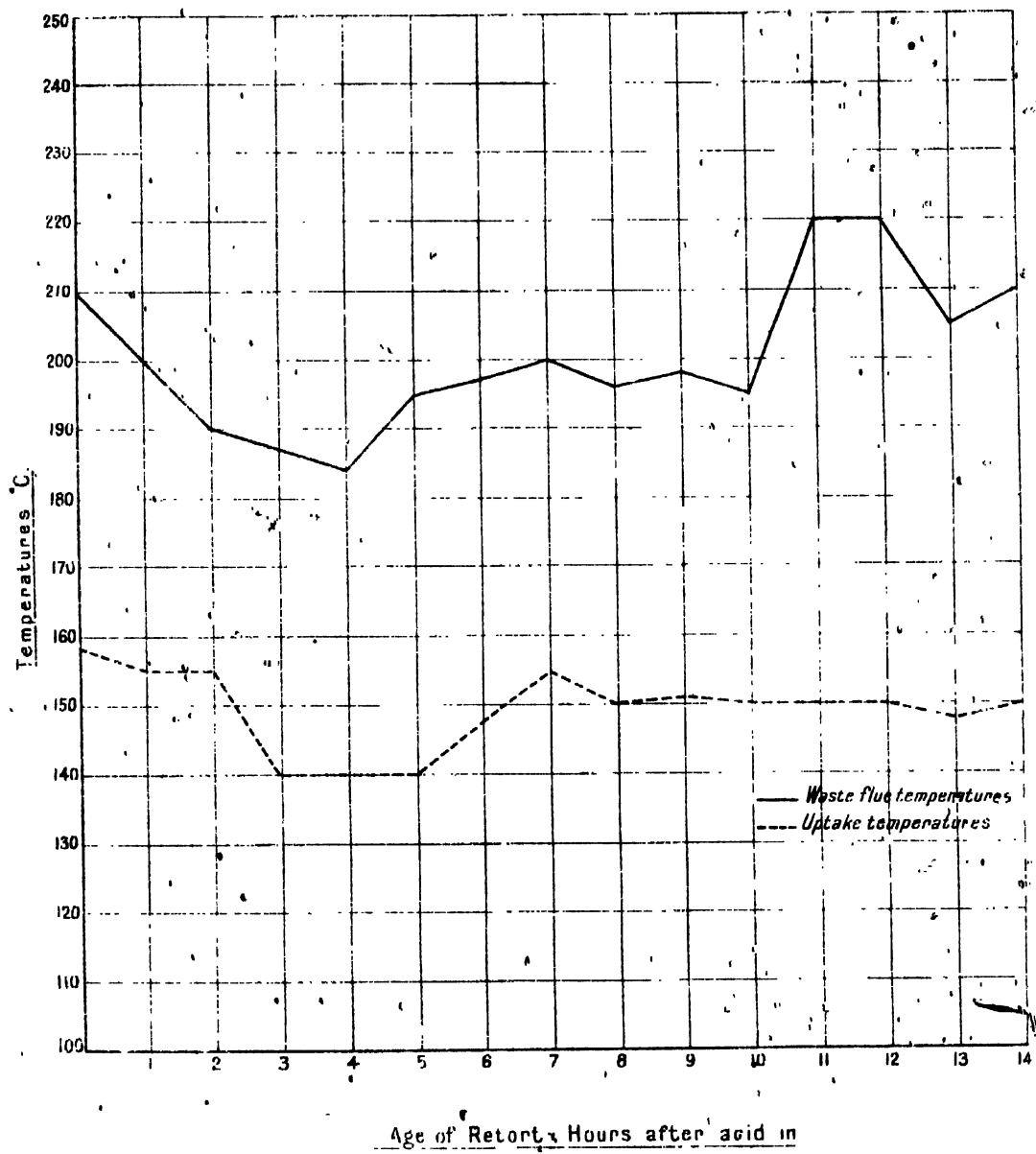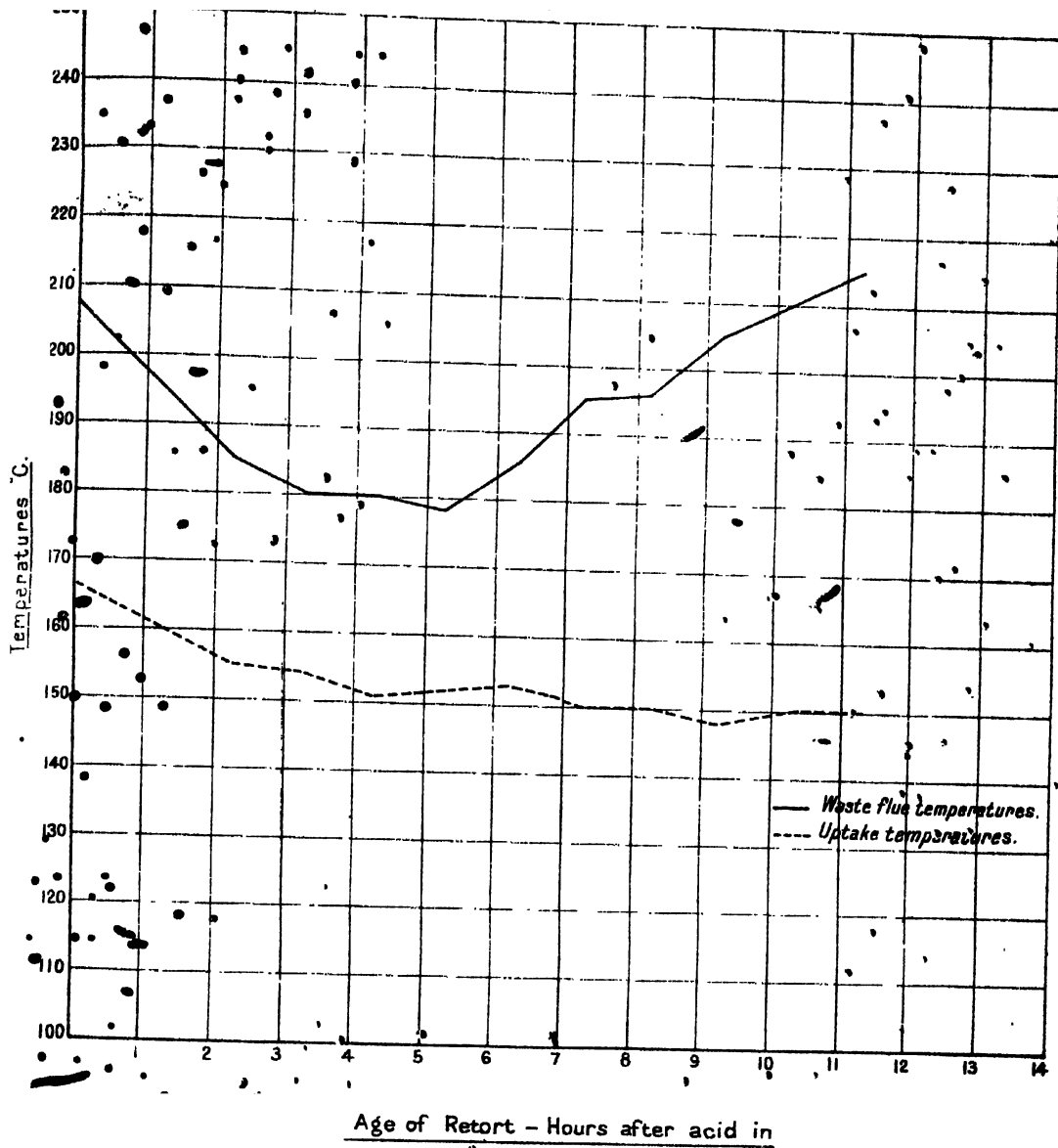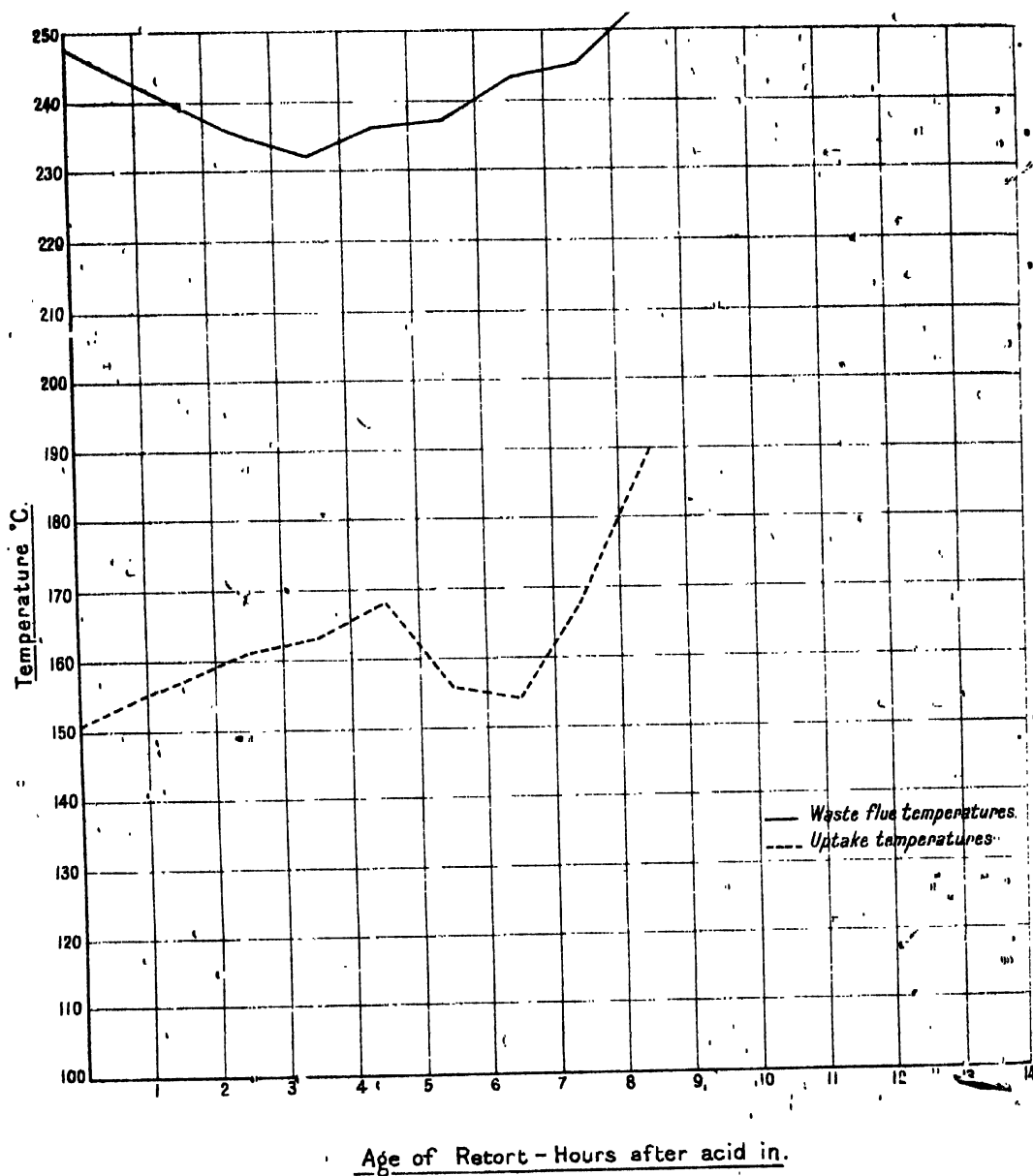
FIG. 28.

and it finishes at a temperature of 190° C.; the highest temperature of the waste flue gases was also recorded immediately before the finish, when it rose to 254° C.

Fig. 26 shows the longest cycle of all, which took 14 hours. These figures were recorded on the cold end of the producer flue, which explains the position.

THERMAL EQUILIBRIUM IN NITRIC ACID MANUFACTURE BY RETORT PROCESS

Heat balance in retort and setting.—This may be conveniently sum-marised as follows : -

A.—Heat absorbed.

Heat taken up by the contents of the retort

(*a*) Heat absorbed in the main reaction.

(*b*) Heating the reacting substance up to the temperature of reaction.

(*c*) Heat required to separate the water from the sulphuric acid added.

(*d*) Evaporation of the water brought in with the nitre and sulphuric acid.

(*e*) Heat of dissociation of a portion of the nitric acid.

(*f*) Heating up the nitre cake from the average temperature of reaction to the final temperature.

II. Heat required to bring up the temperature of retort and setting after re-charging.

III. Heat lost by radiation and conduction through brickwork, top of retort, &c.

IV. Heat carried away by the flue gases.

B.- - Heat supplied.

I. Heat brought in as sensible heat by the producer gas.

II. Heat of combustion of the producer gas used.

The quantity of heat classified as A 1. above may alternatively be considered as made up of the following items :—

(*aa*) Sensible heat in the resulting nitre cake.

(*bb*) Sensible and latent heat in the gases and vapours leaving the retort.

(*cc*) Heat absorbed in raising the internal or intrinsic energy of the reacting substances.

The first classification is, however, much more convenient for calculation.

A.—*Heat absorbed.*

I. (*a*) *Heat absorbéd by the main reaction.*

Heat of formation per gram molecule.

			Cal.			Cal
$NaNO_3$	-	-	- 111	H_2SO_4	-	192·92
$NaHSO_4$	-	-	- 268	HNO_3 vapour	'	34·4
Na_2SO_4	-	-	- 328	HNO_3 liquid		41·6

The main reaction consists of two parts :—

(1) $NaNO_3 + H_2SO_4 = HNO_3$ (vapour) $+ NaHSO_4$
$= (268 + 34·4 - 111 - 193) = -2$ Cal.

(2) $2NaNO_3 + H_2SO_4 = 2HNO_3$ (vapour) $+ Na_2SO_4$
$= (328 + 69 - 222 - 193) = -18$ Cal.
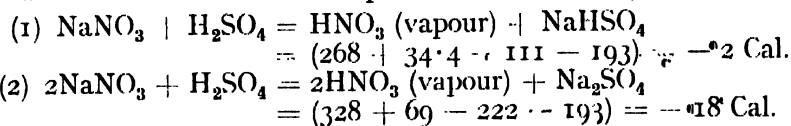
These heats of reaction are calculated on the assumption that the initial and final temperatures are not far removed from $15°$ C., but, as the sum of the molecular heats of the substances on the two sides of, the equation is nearly constant, the heats of reaction at $130°$ to $150°$ will not differ materially from these calculated here.

The nitre cake is stated to contain normally 30 per cent. of " free acid," reckoned as free H_2SO_4.

From the molecular weights $H_2SO_4 = 98$; $Na_2SO_4 = 142$; $NaHSO_4$ = 120, and the equation :—

$$H_2SO_4 + Na_2SO_4 = 2 NaHSO_4$$
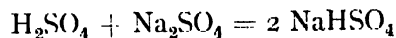
It is seen that 30 parts of " free H_2SO_4 " represent 73·4 parts of NaHSO₄, and that consequently the nitre cake consists of 73·4 per cent. of $NaHSO_4$ and 26·6 per cent. of Na_2SO_4.

The molecular proportions will, therefore, be

$$\frac{73·4}{120} \text{ of } NaHSO_4 \text{ to } \frac{26·6}{142} \text{ of } Na_2SO_4, \text{ or as } 612 \text{ to } 187.$$

In equation (1) one molecule of $NaHSO_4$ corresponds to one molecule of HNO_3, consequently 612 mols. of NaHSO₄ correspond to 612 mols. of HNO_3. According to equation (2) 1 mol. of Na_2SO_4 corresponds to 2 mols. of HNO_3 ∴ 187·5 mols. of Na_2SO_4 correspond to 375 mols. HNO_3.

∴ of the total HNO_3 $\frac{612}{987}$ will be formed according to equation (1),

and $\frac{375}{987}$ according to equation (2).

Hence to produce 63 grams or 1 gram molecule of HNO_3 vapour we require to supply—

$$2 \times \frac{612}{987} + 18 \times \frac{375}{987} = 8·08 \text{ Cal.}$$

or to produce 63 lb. of HNO_3, 8,080 C.H.U. If only 94 per cent. of the total possible HNO_3 is actually recovered, we require to produce 1 ton net—

$$8080 \times \frac{2240}{63} \times \frac{100}{94} = 305,000 \text{ C.H.U.}$$

It will be noted that the heat absorbed by the reaction per ton or per gram molecule of HNO_3 formed decreases with increase of sulphuric acid until the nitre cake contains 100 per cent. of $NaHSO_4$. This variation may conveniently be expressed as a function of the "free sulphuric acid" in the nitre cake. Thus, if H be the heat of reaction per gram molecule of HNO_3 produced, and x the percentage of "free H_2SO_4" in the nitre cake, then

$$H = \frac{1800 - 41 \cdot 1x}{100 - x}$$

where x can vary from 0 to 40·8 per cent., the latter figure corresponding with 100 per cent. $NaHSO_4$. (Higher values of x have no real meaning in this connection.)

Differentiating, we find that—

$$\frac{dH}{dx} = -\frac{2310}{(100 - x)^2}$$

$\frac{dH}{dx}$ lying between $-0\cdot23$ when $x = 0$, and $-0\cdot64$ when $x = 40\cdot8$.

(b) *Heat required to raise the temperature of the initial substances to the average reacting temperature.*

The average temperature of reaction has been taken to be 130° C. and the initial temperature 10° C.

Taking the nitre to be of 96 per cent. purity, and the yield of HNO_3 to be 94 per cent. of the possible, there is required in order to make 1 ton net of HNO_3 :—

$$\frac{85}{63} \times \frac{100}{96} \times \frac{100}{94} = 1\cdot50 \text{ tons of crude nitre.}$$

If 2 per cent. of the crude nitre is water, the crude nitre contains 1·47 tons of dry nitre plus 0·03 ton of water.

The quantity of anhydrous sulphuric acid required to produce 1 ton of HNO_3 with 100 per cent. efficiency is :—

$$\frac{98}{63} \times \frac{612}{987} = 0\cdot965 \text{ ton according to equation (1), and}$$

$$\frac{49}{63} \times \frac{375}{987} = 0\cdot296 \text{ ton according to equation (2), or together}$$
$$1\cdot26 \text{ tons.}$$

But if the sulphuric acid is only of 95 per cent. strength, and the yield of HNO_3 is only 94 per cent., there is required to produce 1 ton net of HNO_3 :—

$$1\cdot26 \times \frac{100}{95} \times \frac{100}{94} = 1\cdot41 \text{ tons of 95 per cent. sulphuric acid.}$$

Taking the following values for the specific heats, viz., $NaNO_3$, 0·278; H_2SO_4 (95 per cent.), 0·35;, and H_2O, 1; the heat required to raise the temperature of the raw materials from 10° C. to 130° C. is for the—

	C H.U.
Dry nitre (1·47 × 0·278 × 2240 × 120) -	110,000
Water in crude nitre (0·03 × 1 × 2240 × 120) -	8,000
95 per cent. H_2SO_4 (1·41 × 0·35 × 2240 × 120) -	133,000
Total - - - - -	251,000

I.—(c) *Heat to separate the water from the 95 per cent. sulphuric acid.*

Since the heats of formation from which the heats of reaction were calculated refer to the anhydrous substances, allowance must now be made for the heat absorbed in separating the water from the 95 per cent. sulphuric acid. 95 per cent. sulphuric acid contains 5·26 lb. of H_2O to 100 lb. of H_2SO_4. From a curve of heats of dilution of sulphuric acid, plotted from a modification of Thomsen's formula, it is seen that the addition of 5·26 lb. H_2O to 100 lb. H_2SO_4 evolves 2,500 C.H.U. Hence to separate the water from 1·41 tons of 95 per cent. acid

$$\frac{1·41 \times 2240 \times 2500}{105·26} = 75,000 \text{ C.H.U are required.}$$

1. (d) *Evaporation of water in the retort.*

1·41 tons of 95 per cent. sulphuric acid contain 0·07 ton of water which, together with the 0·03 ton of water in the crude nitre, makes 0·1 ton. To vaporise this

$$0·1 \times 2240 \times 537 = 120,000 \text{ C.H.U. are required.}$$

I. (e) *Heat of dissociation of nitric acid.*

Assuming that 6 per cent. of the total nitric acid dissociates thus : -

$$4HNO_3 \rightarrow 4NO_2 + O_2 + 2H_2O,$$
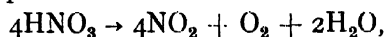
from the heats of formation of the various substances, viz., HNO_3, 34·4; NO_2, 8·3; and H_2O (gas), 57 Cal. per gram molecule, the thermal balance is

$$(2 \times 57 - 4 \times 8·3 - 4 \times 34·4) = -56 \text{ Cal.}$$

or to decompose 252 lb. of nitric acid, 56,000 C.H.U. are required.

If the yield of HNO_3 is 94 per cent., then for 1 ton net recovered,

$$\frac{0·06}{0·94} = 0·064 \text{ ton of } HNO_3 \text{ may be supposed to decompose as above,}$$

and the heat absorbed will be

$$\frac{0·064 \times 2240 \times 56,000}{252} = 32,000 \text{ C.H.U.}$$

It will be noted that the 0·054 ton of nitric acid in decomposing produces 0·009 ton of water vapour. Obviously this must not be included in calculating the heat of evaporation in I. (d); but, on the other hand, it must be allowed for in calculating the sensible and latent heat of the gases leaving the retort.

(f) *Heat required to raise the temperature of the nitre cake from the average to the final temperature of reaction.*

Referring back to I. (a) the theoretical weight of nitre cake per net ton of nitric acid produced is

$$\frac{100}{94} \times \left(\frac{612}{987} \times \frac{120}{63} + \frac{375}{987} \times \frac{71}{63} \right) = 1\cdot 7 \text{ tons.}$$

There appears to be no reliable data for the specific heat of $NaHSO_4$, but it may be estimated from the following values : —

Na_2SO_4, 0·230; K_2SO_4, 0·193; and $KHSO_4$, 0·244

from which, by analogy, the specific heat of $NaHSO_4$ would appear to be about 0·29.

Taking 1·73 tons of nitre cake with an average specific heat of 0·28, the heat required to raise its temperature from 130° to 220° C. is

$$1\cdot 73 \times 2240 \times 0\cdot 28 \times 90 = 98,700 \text{ C.H.U.}$$

II. *Heat absorbed in raising the temperature of retort and setting between charging and discharging.*

To estimate this the heat capacity of the retort and its setting, together with the average temperature range of each, would be needed, expressed relatively to the output of nitric acid per charge.

No data are available.

III. *Heat lost by radiation conduction through brickwork, &c.*

Actual radiation could be most readily determined by a suitable form of thermopile, but air convection, which is probably more important, would be more difficult to measure. A rough calculation could be made from the thickness and thermal conductivity of the brickwork, provided the temperature gradient were known, but exposed portions of the retort would be more difficult to deal with.

IV. *Heat carried away by flue gases.*

To deal with this and the following items it is convenient to combine the figures for the coal consumed in actual practice per ton of nitric acid with the analyses of the producer and flue gases. At the time of the trial, the usual practice was 0·230 ton of coal per ton of HNO₃ produced, whilst the following figures give the composition of the gases :—

Producer gas.		Flue gas.	
	Per cent.		Per cent.
CO	21·0	CO_2 -	1·2
H_2	9·6	H_2O -	·9*
CH_4	1·8	$O_2 + N_2$	·9*
CO_2	7·0		
N_2	60·6		
	100·0		100·1

* * Re-calculated to agree with percentage of CO_2.

15092

Heat value per cubic foot $=$ 111·4 B.T.U. or 61·9 C.H.U.

It is of interest to compare the composition of an ideal flue gas, produced by combustion with the theoretical amount of air, with that of the actual flue gas.

100 vols. of producer gas	Air for each constituent	
	Per cubic foot.	Per vol. present.
CO 21·0	2·4	50·4
H₂ 9·6	2·4	23·0
CH₄ 1·8	9·6	17·3
CO₂ 7·0	—	—
N₂ 60·6	—	—

The total air is therefore 90·7 volumes, which contain 71·7 volumes of N_2. The composition of the ideal flue gas is, therefore,—

Per 100 vols. of producer gas—	Composition per cent. of ideal flue gas—	
	As existing in flue.	As measured in laboratory.
CO₂ 29·8	17·0	18·4
H₂O 13·2	7·5	} 81·6
N₂ 132·3	75·5	
175·3	100·0	100.0

From the composition of the gases and recorded values of specific heat and density the following figures are readily obtained :—

$$1 \text{ gram/litre} = 0.0624 \text{ lb./cubic feet.}$$

	Producer gas.	Flue gas.	
		Ideal.	Actual.
Density lb./cu. ft.	0·074	0·083	0·031
Specific heat 0°–200°	0·24	0·25	0·24

Taking 135,000 cubic feet of producer gas per ton of coal and 0·230 ton of coal per ton of HNO_3, one ton of nitric acid requires 31,100 cubic feet of producer gas, which, with the ideal amount of air, should give 54,300 cubic feet of flue gas containing 17·0 per cent. of CO_2. The actual flue gas, however, contains only 4·2 per cent. of CO_2, so that its volume must be 220,000 cubic feet.. That is to say, three-quarters of the actual flue gas is excess air—a very poor result for gaseous fuel.

If the average temperature of the flue gas is 200° above atmospheric, the heat carried off with ideal combustion would be

$$54,300 \times 0.083 \times 0.25 \times 200 = 225,000 \text{ C.H.U.}$$

whilst in practice it is

$$220,000 \times 0.081 \times 0.24 \times 200 = 855,000 \text{ C.H.U.}$$

namely a partially avoidable loss of   630,000 C.H.U.

B.—*Heat supplied.*

I. Sensible heat in the producer gas.

The temperature of the incoming producer gas is given as about 300° C. above atmospheric. The heat brought in is therefore

$$31,100 \times .074 \times 0.24 \times 300 = 166,000 \text{ C.H.U.}$$

II. Heat of combustion of producer gas.

This is $31,100 \times 62 = 1,930,000$ C.H.U.

Summary of heat balance of retort and setting.

So far as the available data permit, the heat balance may be shown as follows :—

A.—*Heat absorbed.*

	C.H.U.	C.H.U.
I. Absorbed by contents of retort.		
(a) By the main reaction	305,000	
(b) In raising temperature of raw materials to average temperature of reaction	251,000	
(c) Separation of water from the sulphuric acid	75,000	
(d) Evaporation of water in the nitre and sulphuric acid	120,000	
(e) Dissociation of portion of the nitric acid	32,000	
(f) Raising the temperature of the nitre cake from the average to the final temperature	102,000	
		885,000
II. Heat absorbed in raising the temperature of retort and setting between charging and discharging		
III. Heat lost by radiation conduction through brickwork, &c.		
IV. Heat carried away by flue gases		855,000
Balance to be accounted for		356,000
Total		2,096,000

B.—*Heat supplied.*

	C.H.U.
I. *Sensible heat in the producer gas*	166,000
II. *Heat of combustion of producer gas*	1,930,000
Total	2,096,000

Reverting to the alternative method of treating the heat absorbed by the contents of the retort, mentioned on pages 1 and 2 of these notes, the items A I. (*aa*) and A I. (*bb*) should be considered.

A I. (*aa*) *Sensible heat in the discharged nitre cake.*

Given 1·73 tons of nitre cake with specific heat 0·28 at 220° C. (say, 210° above atmospheric temperature) the sensible heat per ton net of HNO_3 is

$$1·73 \times 2240 \times 0·28 \times 210 = 228,000 \text{ C.H.U.}$$

A I. (*bb*) *Sensible and latent heat in vapours leaving retort.*

	C.H.U.
Take the value for the nitric acid vapour	355,000
and for the accompanying steam	196,000

In addition the 0·064 ton of nitric acid which is decomposed (*see* A I. (*e*)) will yield 0·055 ton of NO_2 and O_2, the sensible heat in which will be

0·055 × 2240 × 0·24 × 210 or about	6,000
The total amount under this head is therefore	557,000

which represents roughly the amount of heat which must be absorbed by the condensers per ton net of HNO_3 recovered.

Remarks.

The producer gas was of poor quality. The percentage of CO_2 in the flue gas being only 4·2 indicates that far too much air was being used. As shown before, with ideal combustion 100 volumes of producer gas would require only 90·7 volumes of air, yielding a flue gas with 17 per cent. of CO_2. Since only 4·2 per cent. of CO_2 was present the volume of flue gas per 100 volumes of producer gas must have been 709 volumes, of which 534 volumes were excess air. The total air used was therefore 624 volumes instead of 90·7 volumes, or about seven times as much as was ideally necessary. Hence the excessive loss of heat in the flue gas.

In considering the thermal conductivity of the walls of a condenser, note should be taken of the adhering layer of poorly conducting liquid. This layer will tend to be thinner the steeper the fall in the condenser tubes.

In comparing the Hart condenser with the spiral silica condenser, it is possible that for equal surface a spiral is more efficient than a straight

tube, since the continual change, in direction of flow may be expected to bring about a better circulation of the vapours in the tube.

Thermal efficiency of condensing plant: Comparison of Hart and silica sets

At *Oldbury* the silica sets comprised two 5 inch off takes supplying four silica coils (2 inch diameter).

At Queen's Ferry—One 8 inch off take branches into two 6 inch pipes and each pipe supplies 26 (1¼ inch) glass tubes.

		Offtake.	Condensers.
Silica sets	-	- 50 k	$4 \times (2)^2 k = 16 k$
Hart sets	-	- 64 k	$2 \times (6)^2 k$ leading to
			$52 \times (1\frac{1}{4})^2 k = 81 k$.

Where k is a common constant.

Silica set is throttled down from 50 to 16.
Hart set opens out from 64 to 81.

Efficiency of Hart condensers— Queen's Ferry.

Each retort is fitted with 78 glass tubes, each 6 feet 7 inches in length, and 1¾ inch average external diameter. Total cooling surface $= 78 \times 6.58 \times 3.1416 \times 0.115 = 186$ square feet. 1.65 tons of HNO_3 as vapour, carrying 0.138 ton water vapour for each ton HNO_3, requires $(1.65 \times 557,000)$ C.H.U. to be abstracted from it in order to obtain liquid at 15° C. in eight hours.

Heat to be taken up in condensers

$$= \frac{1.65 \times 557,000}{8 \times 60 \times 60} = 32 \text{ C.H.U. per second.}$$

$$\frac{32}{186} = 0.172 \text{ C.H.U. per square foot of cooling surface per second.}$$

$$= \frac{0.172 \times 453}{929} = 0.0839 \text{ cal. per cm.}^2 \text{ per second.}$$

Thickness of tubes $= 1.16$ inch $= 0.159$ cm.

Conductivity of glass (soda flint glass) $= 0.00143$ cal./sec./1° C./cm.^{2}.

Heat which will pass through tubes at unit temperature gradient

$$\frac{0.00143}{0.159} = 0.0090 \text{ cal. per square cm. per second.}$$

Heat required to pass for effective working $= 0.0839$ cal. per cm.^{2} per second,

Hence temperature gradient required $= \frac{0.0839}{0.009} = 9.3° C.$

Efficiency of silica condensers, Oldbury.

Each retort is fitted with four silica coils, 7 inches internal diameter, 5/32 inches thick, diameter of coil 2 feet 6 inches, $7\frac{1}{2}$ turns. Condensing surface (external) $= 4 \times 2 \cdot 5 \times 3 \cdot 14 \times \dfrac{(2 \cdot 3125 \times 3 \cdot 14) \times 7 \cdot 5}{12}$

$$= 143 \text{ sq. ft.}$$

Assume working as at Queen's Ferry :—

Then heat taken up — 32 C.H.U. per second.

$$\frac{32}{143} = 0 \cdot 224 \text{ C.H.U. per sq. ft. per sec.}$$

$$\frac{0 \cdot 224 \times 453}{929} = 0 \cdot 109 \text{ cal. per sq. cm. per sec.}$$

Conductivity of silica (quartz) $= 0 \cdot 016$ cal. ser. 1° C. cm.^{2}

Heat which will pass through coils ($5/32$ inches $= \cdot 397$ cm.) at unit temperature gradient $= \dfrac{0 \cdot 016}{\cdot 397} = 0 \cdot 0403$ cal. per sec. per cm.^{2}

Heat required to pass — $0 \cdot 109$ cal. per sec. per cm.^{2}

Temperature gradient $= \dfrac{0 \cdot 109}{0 \cdot 0403} = 2 \cdot 7$ C.

The four silica coils have a cooling surface of 143 square feet.

The Hart set has a cooling surface of 186 square feet.

Owing to the greater conductivity of the silica the temperature gradient is only $2 \cdot 7$ compared with $9 \cdot 3$ for the glass tubes in the Hart set.

CRACKING OF RETORTS AND STILLS

At one time considerable trouble was caused by cracking of many of the retorts. The plants were being worked very intensively and the cracking was considered to be due in many cases to bad firing.

As first erected, the bottom of the retort rested on a small brick slab directly on top of a narrow fire arch. Afterwards the fire arch was lowered and broadened so that the flame did not strike the bottom of the retort directly, but was deflected and spread by the arch, also when the fire was put out the retort cooled quicker as it was not kept so hot by being in direct contact with the fire arch. This arrangement helped to prevent cracking.

No definite conclusions could be drawn as to the composition of the cast iron having anything to do with liability to cracking. More importance should probably be given to the foundry practice when casting the retorts.

It was often possible to prolong the life of a cracked retort by tapping holes round the crack and screwing on a cast iron patch making a joint between the patch and retort with rust and sal ammoniac.

The following figures from the records of one factory give an indication of the average life of a retort or still :—

(1) The average number of charges of stills and retorts which have failed.

(2) The average *maximum* number of charges of a similar number of stills and retorts which up to the present have not failed.

(3) The average life has then been taken as the mean of these two averages.

Basing the estimate of the average life of a still or a retort on the above assumptions, we find the following :—

Stills.

Average No. of charges of six stills which have failed - - - - - - -	270 charges each.
Average No. of charges of six stills which are still operating and have not shown signs of failure	365 charges each.
Mean average of the above which should represent the average life of a nitric acid still. (Each charge being 7 tons of mixed acid) - -	317 charges.

Retorts.

Average No. of charges of 12 retorts which have failed .- - - - - - -	175 charges each.
Average No. of charges of 12 retorts which have not failed and are still being operated	355 charges each.
Mean average of the above which should represent the average life of a retort. (Each charge containing 2 tons of nitre) - - -	265 charges.

A large American factory reported that while 400 charges might be taken as the average life of a retort, individual retorts had stood 800–1,000 charges.

At another factory (not American) with an installation of 75 retorts, one retort has been in use over 11 years and 30 are over 10 years old.

Skilled firing and not too intensive working combined with good construction of the combustion chamber have much to do with the long life of retorts.

SECTION 2

NITRIC ACID STILLS FOR DISTILLATION ON SPENT ACID FROM THE MANUFACTURE OF NITROCOTTON

DISCONTINUOUS DISTILLATION

General outline.—The spent acid is run into a cast-iron still heated with a gas fire, and the nitric acid in the spent acid distilled over into a condensing system. The residual sulphuric acid in the still is removed to the concentration plant.

The plant used for this purpose is similar to that just described for making nitric acid. The chief difference being in the still which has no opening at the bottom (Fig. 29).

When using the distillation process for denitrating spent acid, it is essential that the composition of the acid charged to the stills be such that the residual acid after all the nitric is driven off contain not less than 73 per cent. of H_2SO_4, otherwise the nitric acid will be too weak.

Consequently it is often necessary to add a suitable quantity of strong sulphuric acid to the spent acid to ensure a sufficiently strong sulphuric acid being left in the stills, which will keep the greater part of the water from being driven over with the nitric acid.

Description of plant.—Spent acid storage consists of eight 30 ft. × 9 ft. mild steel cylindrical boiler tanks, built upon brick piers, above the level of the stills in the house, so that all charging of stills with acid is by gravitation. The boilers are inclined so that one end is 2 inches lower than the other, to allow sludge to fall towards the sludge-outlet. At the opposite end, and on the floor of the boiler, is the acid outlet, into which fits a cone plug controlled by a lever and chain. The run-off is supplied with a Quinan valve, and is connected to a 3 inch mild steel acid main. Each boiler is fitted with a gauge glass, and is capable of holding 89 tons of spent acid of specific gravity 1·680. On the top of the boiler is a man-hole, surrounded with a tripod supporting a dipping rod. At a depth of 6 feet 6 inches an overflow pipe runs common to all the boilers, and similarly a common 3 inch main is fitted at the bottom through which each boiler can be emptied. The 3 inch connecting main feeds a main of the same diameter, which runs the whole length of the still house. From this latter each still is charged.

There are 30 stills in the house, arranged in batteries of three. Each still consists of a cast-iron cylindrical vessel with a spherical bottom. The body is 2 inches thick and the bottom 3 inches thick

				Ft.	in.
Total depth of still	-	-	-	- 7	0
Diameter of still -	-	-	-	- 6	9
Width of lid -	-	-	-	- 3	4
Width of man-lid	-	-	-	- 1	8

Each still is completely enclosed in a brick setting.

The dome cover has a flattened top, and is fixed to the pot by means of a rust joint. The lid is bolted on to the top of the dome cover. The lid is recessed to a depth of 2½ inches and 12 inches diameter round a circular hole 8 inches in diameter. Into this 8 inch hole is fixed the still head. The man-lid is perforated with a hole 3½ inches in diameter, through which passes a 7 foot dip pipe reaching to the bottom of the still, and also with a ½ inch hole in which an air pipe is fixed.

The still takes a charge of 7 tons of spent acid, and when in continuous use, the nitric acid can be distilled off in nine hours. The complete cycle from starting one charging to starting the next charging is 12 hours, so that two charges per day of 24 hours can be put through.

Producer gas is used for firing in a similar manner to the retorts, and the heating of the still can be controlled within very fine limits.

The charging and blowing pipe is of lead, 2½ inches in internal diameter, and conveys the spent acid from the charging main to the still, and also delivers the residual acid, at the end of the distillation, into the launder leading to storage of weak sulphuric acid.

The connection between this pipe and the 3 inch feed main containing spent acid consists of a 1½ inch lead " U " bend, with two plug cocks situated in front of the latter. The charging and blowing pipe is supported on channel iron, and is continued out of the house to the launder mentioned above. Between the launder end of the charging and blowing pipe and the inlet from the " U " bend is placed a 3 inch plug valve, which is always closed during distillation and charging.

The other end of the charging and blowing pipe is bolted to a swan-neck connection of the " dip " pipe in the still.

The dip pipe is made of cast-iron or silicon-iron. The latter, though more costly in initial outlay, is finally cheaper than cast-iron, because of its much longer life. A cast-iron dip-pipe very easily chokes with sulphate, and corrodes rapidly.

Length of pipe	-	-	-	-	7 feet 2 inches.
Internal diameter	-	-	-	1½ inches.
External diameter	-	-	-	3 inches.

The still is charged through this pipe, and the contents at the end of distillation are discharged through it into an open semi-circular lead launder, suspended in water contained in a wooden trough, which conveys the acid to the weak sulphuric acid storage tanks which it reaches with a temperature of 70°–80° C.

Condensing system. The still head is a two-way piece with an internal diameter of 8 inches and made of silicon-iron. It is fixed to the 8 inch opening in the lid of the still with cast-iron collar plates bolted down at its base. The vapours pass up into the still head, and are shunted through the bend into the uptake. The short vertical extension of the still head is used for dipping, and to allow the fixing in of the sealing plug when the contents of the still are ready for discharge at the end of the distillation.

The sealing plug is a flat disc made of cast iron. It has an elevated rim on its circumference, which fits into a groove running round inside

the bottom of the still head. The object of this disc is to seal completely the vapour outlet from the pot, in order to allow the residual acid to be blown out of the still by means of compressed air. The acid is blown back through the dip pipe into the launder. The compressed air is allowed to enter the still through a small $\frac{3}{4}$ inch pipe screwed into the man lid. The supply of air to the stills is brought from a common air main, running the whole length of the house, through individual 1 inch pipes. The pressure of air in the common main is kept at 10 lb. per square inch, being reduced to this from 80 lb. per square inch in the main line.

The uptake is connected with Hart condensers and absorption towers in the same way as in nitric acid manufacture from nitre.

Operation of the plant.—The spent acid to be distilled has an average sp. gr. of 1·680 at 15° C., and is of a pale straw colour, gives off nitric acid vapour on exposure to air, and contains a slight amount of sludge which is chiefly ferric sulphate. Its average composition by weight is—

		Per cent.
HNO_3	- - - - - -	20·3
H_2SO_4	- - - - - -	60·9
H_2O	- - - - - -	18·8

Its action upon mild steel, lead, or cast iron at ordinary temperatures is not excessive, and it can be stored in boiler tanks of mild steel with safety.

The storage of the spent acid is at such a height that all stills can be charged by gravitation. The storage boilers are sludged periodically, and kept well painted. All valves, both inlet and outlet, are kept well greased, and turned at least once every day. The staging and steps around the storage boilers are well painted, and also well illuminated for dipping &c., during the night.

A gangway runs across from the charging platform of the house, and in the event of valve trouble, &c., speedy communication is possible. From the dimensions of the pot the tonnage or capacity is found, and on this plant a charge of 4 feet 9 inches of spent acid is approximately seven tons by weight. The charging valve is opened, and the acid runs in with a force dependent upon the head. Twenty minutes may be taken as the average time required to run in a charge. The pot is dipped at intervals with a copper rod, having a ring marked at a point 4 feet 9 inches from its bottom end. The rods are checked in measurement once every week.

Iron rods have been tried, but overcharging frequently occurs by reason of " sweating " on the surface when the rod is put into the still, which gives a very indistinct mark. Of late, an electrical charging appliance has been used, which measures the charge with a " dry " dip and rings an alarm bell when the still is fully charged.

When the charge has been run in, the still is sealed up at the still head in readiness for distillation. Two stills can be charged at the same time, but then the charging time is lengthened from 20 to 30 minutes.

After distillation is complete, the fire is put out and the cover from the still head is removed. The blowing plug has been prepared, and this is immediately screwed down into position by means of a screw clamp. In preparing the blowing plug, it is necessary to provide it with a suitable packing which will withstand the temperature and the acid fume, and it has been found that blue asbestos cord is the best for this. One piece, if properly handled, is sufficient for 20 or more blows. When the plug is in position, the air pipe is connected up to the still and compressed air turned on.

The air pressure used is 10 to 11 lb. per square inch, and the supply should be perfectly free and steady. The dip pipe should be clear of sludge. As the pot empties, the rate of discharge of the residual acid diminishes somewhat, due to increased lift as the acid level in the pot lowers.

The fire may be put on when the still is half charged, and a good brisk fire kept on until distillation commences, which is, on the average, 2½ hours afterwards. The fire is lessened somewhat when distillation has commenced, but every care should be taken to avoid decrease of temperature. It will often be necessary to increase the fire towards the end of the distillation rather than to reduce it. Without good firing good results cannot be obtained, and it is necessary to give the fire every care, especially so with a view of getting good fractionation. A slow inactive fire gives very unsatisfactory distillate. Large quantities of nitrous acid are formed, and the distillation becomes unduly lengthened.

Vapours begin to pass over at 90° C., containing much of the nitrous acid contained in the spent acid. At 135° C. a good distillate is obtained, and it is at this point that most of the HNO_3 is obtained. The temperature of the vapours passing up through the uptake gradually increases, and the HNO_3 content gradually decreases until, at a temperature of 145°–150° C., the distillation is complete. This point, however, is not determined by temperature, but by taking the gravity of the distillate. When the gravity of the distillate has fallen to 1·300 the distillation is complete.

At present the whole of the distillate from a charge is run into the same receiver, and the resultant is an acid of average strength of 89·5 per cent. HNO_3. Each 7 ton charge of spent acid gives approximately 1·4 tons of HNO_3.

The control of the condensers is an important item, and care is especially required in the water feed. Spent acid from cotton nitrations contains an appreciable amount of nitrous acid. It is therefore more economical to condense HNO_3 only in the condensers, and allow the nitrous acid to pass on to the absorption system. This can be controlled on the condensers only, and is of much importance. To carry out successfully the elimination of nitrous acid to the maximum, it is necessary to run the condensers as hot as possible, consistent with efficient condensation of the nitric acid throughout the whole distillation. More water is required on the condensers in the middle of the distillation than at the beginning or the end.

All gravities of the distillates are taken on the still-watcher, or gravity platform, which runs directly underneath the condenser platform. The " gravity-man " informs the fireman when a still has finished. Preparations are then made to " blow " the still and clean the gas valve.

As soon as a receiver is filled, a sample is drawn for analysis, but it is essential that the contents of the receiver be well agitated with compressed air before being sampled. This serves a double purpose, in that it removes a little nitrous acid and mixes the contents. Before emptying a receiver the gravity is taken.

Owing to the much greater amount of oxidisable nitrogen compounds entering the stills absorbers, as compared with those of the retorts, a somewhat different method of control is required. The Pohle air·lift works much faster, and ensuing oxidation reaction is more intense, and the yield of HNO_3 considerably greater. The gases enter during the summer months with an average temperature of 30° C., and, by the time they have reached the third tower in the set, the temperature has risen to an average of 50° C. The make of acid in the towers runs between 9 per cent. and 10 per cent. of the total HNO_3 made, and it contains on the average 0·1 per cent. of nitrous acid.

Working results during one month.

Stills charged 1,321.
Average number of stills working, 22.

Spent acid used $= 9040·3$ tons at 20·3 per cent. HNO_3
$= 1836·8$ tons HNO_3

Plant loss.

Nitric acid charged $= 1836·8$.
Nitric acid recovered $= 1788·4$.
Recovery $= 97·4$ per cent.
Strong nitric acid produced $= 1784·1$ tons at 90·0 per cent. HNO_3
Weak nitric acid produced $= 351·8$ tons at 52·1 per cent. HNO_3
Strong nitric as $HNO_3 = 1605·7$ tons.
Weak nitric as $HNO_3 = 183·0$.

Hours of distillation, 6 hours 30 minutes.
Total cycle, 10 hours 40 minutes.

Labour requirements.

For complete running of 26 stills, allowing four out of commission for repair, the following are necessary :—

> Foreman
> Charge hand
> 6 male operatives } per shift.
> 2 burner men

For maintenance of pottery, four men on day work. In addition, plumbers, fitters, and joiners, &c. are required at intervals.

FIG. 30.

To Nitric Receivers.

HNO₃ 90%
HNO₂ 2·5%

Still Watcher.

One Coil only shewn.

116°C.

Fume Main.

Cast Iron

Pottery

to Flue 430°C.

Packed Graded Quartz.

8 tons - 24 hrs.
H₂SO₄ 61%
HNO₃ 19½%

Feed Acid.

D.N.A. 75% H₂SO₄ 175°C.
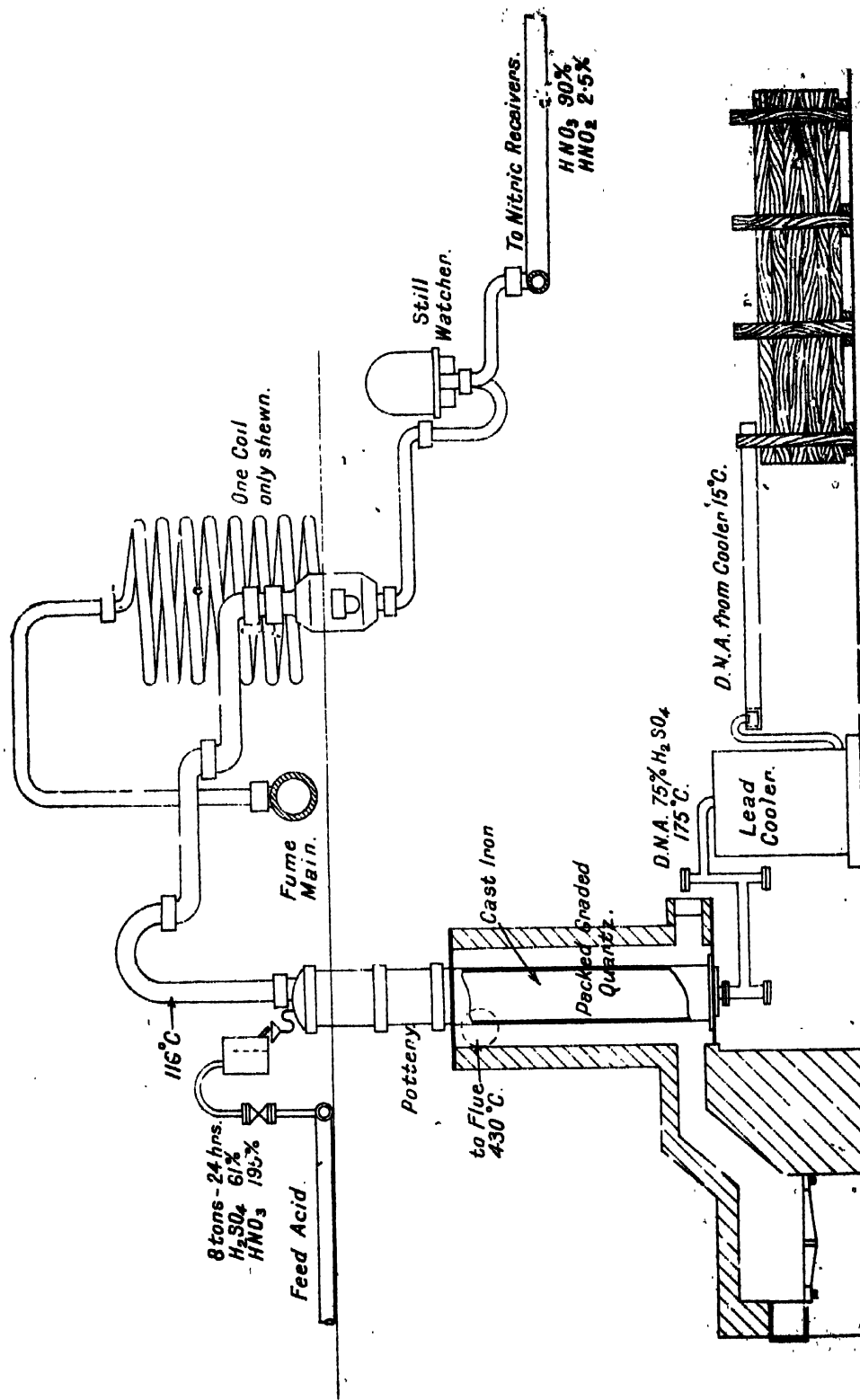
Lead Cooler.

D.N.A. from Cooler 15°C.

Mailby & Sons Lith.

CONTINUOUS DISTILLATION

Some trials may be recorded of a continous distilling or dephlegmating column which were stopped owing to the Armistice before the best working conditions were ascertained or conclusive results obtained. They promised well and should be worth following up. The cast-iron column is very small and inexpensive for the work performed, and can be quickly and readily replaced when necessary.

A small continuous distilling or dephlegmating column was erected and tried at Queen's Ferry, but the experience in working it was too limited to allow definite conclusions being drawn as to its advantages or disadvantages in comparison with the ordinary still working continuously.

Description of plant.—The original dephlegmating column, of which a diagram is shown (Fig. 30), consists of a cast-iron column 8 feet high × 14 inches inner diameter, on top of which are fitted two pottery sections, each 14 inches internal diameter × 2 feet high. It was necessary, at the very outset, to replace these by silicon-iron sections, owing to the cracking of the pottery.

The column was packed with carefully graded quartz, the packing resting on a cast-iron perforated plate. This corroded very speedily, however, and was replaced by a grid of acid proof pottery rings.

Heating was supplied by an oblong coke combustion chamber, from which the hot gases were led to a brick chamber 2 feet 3 inches square and 7 feet 7 inches high, surrounding the cast-iron column, the waste gases being led away to the main flue by a 10 inch diameter mild steel pipe.

The earthenware cover to the column was provided with a 4 inch off-take for the nitric acid vapours and two 1 inch holes, one being for the spent acid feed and the other for manometer and thermometer.

A 2½ inch outlet from the base of the cast-iron column delivered, through a silicon iron seal-pipe into a circular acid cooler, similar to those at the TNT acid denitrating plant,* one cooler being provided to each pair of columns. The coolers deliver into a lead launder and thence by gravity into either of two storage tanks, from which the acid is pumped to the battery of lead denitrated acid tanks for acid from the still.

The spent acid is fed into the top of the column through a pottery feed box delivering into a 1 inch lead luted feed box. The distribution of the acid in the column is an important point, as it is essential to counteract any tendency for the acid to flow merely down the centre of the packing.

The nitric acid vapours are led through the 4 inch diameter outlet to silica coils in exactly the same way as described in the case of the stills.

Capacity of plant.—As far as the limited number of experiments made seemed to point out, the capacity of a single column was in the

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* See No. 1 Record of this series.

neighbourhood of about 5 tons of spent acid per day. This was increased to 7 tons per day, and a considerable saving of fuel effected, by lagging the whole exterior of the furnace and the flue surrounding the cast-iron columns.

The fuel consumption was abnormally high. The temperature of the gases in the off-takes from the flue surrounding the column had to be as high as 400° C. to ensure thorough denitration. This represents very considerable waste of heat, which might be reduced to some extent by extending the length of the column or, at any rate, the length exposed to the heating effect of the gases.

Comparison of stills and dephlegmators.—The working of the stills and dephlegmators is compared in the following table :—

	Still.	Dephlegmator.	
		Lagged.	Unlagged.
	Tons.	Tons.	Tons.
1. Capacity per day for complete denitration - - - - - -	9–10	7	4–5
2. Strength of denitrated acid, 61·0 : 18·5 : 19·5 and spent acid - -	H_2SO_4 77·5 per cent.	H_2SO_4 75·76 per cent.	H_2SO_4 75·76 per cent.
3. Strength of strong nitric acid :			
HNO_3 - - - - - -	89 per cent.	90 per cent.	90 per cent.
HNO_2 - - - - - -	1·1·7 per cent.	2·3 per cent.	2·3 per cent.
4. Fuel consumption per ton HNO_3 recovered - - - - -	0·22 ton coal	0·75 ton coke	1·4 tons coke

Trials were also carried out at Gretna with a continuous distilling column similar to the one used at Queen's Ferry.

The column was 12 feet in height, 14 inches diameter, and was packed with graded quartz. The top 4 feet of the column was of silicon-iron, and the fume outlet was increased to 8 inches in diameter.

The results obtained were very promising.

When treating nitrocotton spent acid, a stronger distillate and weaker residual acid were obtained than from the ordinary still.

About 12 tons of spent acid could be treated per day per column. No reliable fuel consumption figures were obtained.

Experience showed that the most suitable temperature was 100° C., and, when working under this condition, the following acids were obtained :—

Condensate.

Specific gravity, 1·520.

		Per cent.
Total nitrogen acids as HNO_3		102·00
„ „ HNO_3		97·80
„ „ HNO_2		3·09

Residual acid.

Specific gravity, 1·700.

Nitrogen acids - - - - Trace
H_2SO_4 - - - - - - 76·00 per cent.

This temperature appeared to be the most suitable for the column. Trials made at higher temperatures produced a weaker condensate and a stronger residual acid.

When distillation was attempted at a lower temperature the acid issuing from the base of the tower contained quantities of nitric acid.

Throughout the whole of the experiments difficulty was experienced with sludge. The base of the column required clearing about once every 24 hours, but steps were taken to overcome this by modifying the base of the column.

SECTION 3

DESCRIPTION OF PLANT AND PROCESS FOR WASHING NITRE BAGS

General outline.—This plant was designed for the purpose of treating bags 'in which nitre had been brought into the factory with the object on the one hand of yielding dry bags as free as possible from nitre, and, on the other hand, of recovering as much as possible of the nitre removed from the bags in a form suitable for the manufacture of nitric acid.

The following is a description of a typical bag-washing plant, as first designed. Experience proved later that various modifications might be made which not only gave better results but also simplified the process.

Dirty nitre bags are first brushed to remove all loose nitrate. They are then boiled up in dissolving tanks. The liquor obtained when sufficiently concentrated is lifted by means of two blow eggs to a weak liquor tank from which it is run through an evaporator to a crystallising tank. The bags are passed through four rinsing tanks, wrung in centrifugals, dried in steam-heated driers, and finally sorted out according to quality.

Description of plant. The plant (Fig. 31), which was designed with a view to the treatment of 5,000 bags per day, consists of three separate brick buildings, viz., the dirty bag store, the washing plant (which may be looked upon as the main building), and the clean bag store.

Dirty bag store. This is situated near the end of the washing plant, where the boilers are placed. The buildings stand on a platform, about 3 feet 6 inches above ground level, which runs into the main building, and on which the boiling tanks stand.

'Trucks of bags are brought alongside the platform on rail, and discharge direct into the store. The bags are turned inside out and brushed and the loose nitre sifted to separate string. This nitre is then 'bagged and returned to the nitre store. The brushed bags are taken in trolleys to the washing plant. Trolley rails lead from the bag store into the main building to the back of the boiler tanks. Originally the bags were brushed by hand, but a mechanical brusher has since been installed. This has proved both cheaper and more efficient than hand brushing.

The capacity of the store is about 15,000 bags.

Washing plant.—The washing plant in the main building consists of

1. Boiling tanks.
2. Troughs.
3. Centrifugal extractors.
4. Driers.
5. Eggs, filter presses and reservoirs.
6. Evaporators.
7. Crystallising pans.
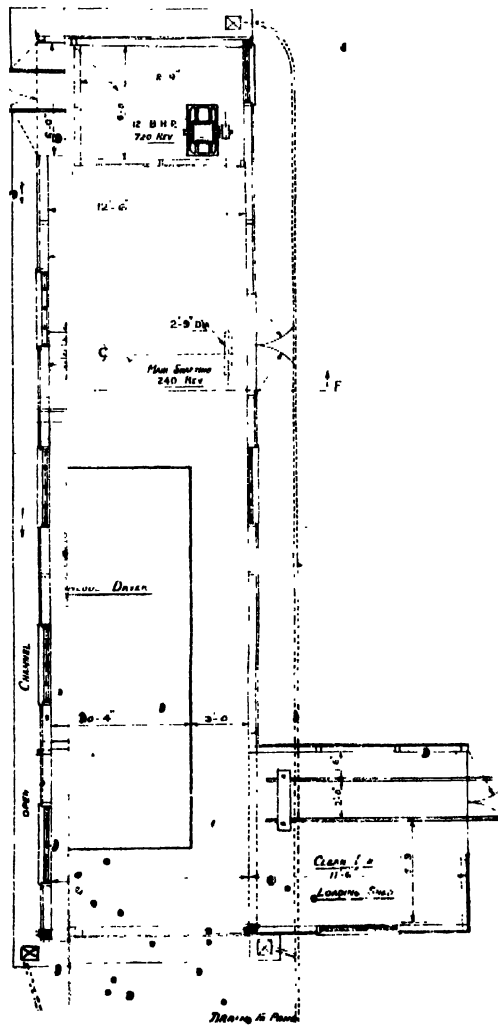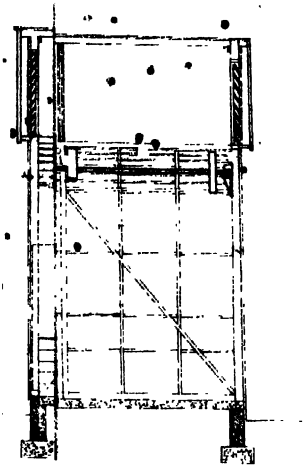
Boiling tanks.—These are three tanks in which the bags are brought in contact with water containing more or less nitre in solution, and heated by the introduction of steam. The tanks are cylindrical in shape and each contains a sheet-iron grid or basket for the bags, perforated with 2 inch holes and supported 2 inches away from the bottom and sides of the boiler, so that the bags are at all times completely surrounded by liquid. The steam pipe leads to the bottom of the tank and then extends in three lengths across the bottom, so that the heating is evenly distributed over the bottom. The capacity is 16 cubic feet. A pneumatic ram is fitted above the tanks for pressing the bags into the liquor and for expressing contained liquor from the bags after the liquor has been run from the tank. There are two bottom outlets in each tank connecting with the blow eggs for delivering the liquor after extraction.

Troughs.—There are four wooden troughs or vats through which the bags pass in succession after leaving the boiling tanks. Each is 11 feet 1 inch × 3 feet 4 inches × 2 feet 3 inches internal dimensions. The trough farthest from the boiling tanks is fed by a fresh-water tap, and the water passes back from the trough (No. 4) through the other three to the boiling tanks in a direction counter to that of the bags by means of wooden launders situated at one end of and near the top of each trough.

In the first three troughs a wooden launder runs along the length of the trough on the water exit side, and this is fed from the bottom of the trough by a shaft, so that the liquid running from, say, trough 3 to trough 2, is drawn from the bottom of trough 3. Trough 1 is connected to the boiling tanks by an iron pipe from the bottom of the trough with three branches, one to the bottom of each tank. By opening a valve in the branch pipe the liquid from trough 1 runs into the tank. The level of the liquid in trough 1 is maintained by keeping up the level in trough 4 by means of the tap.

As the bags leave the boiling tanks they are placed on draining boards slightly inclined backwards so that any liquid dripping off them runs back to the tanks. Similar draining boards are interposed between each pair of troughs and at the end of trough 4.

Centrifugals.—There are two centrifugal patent self-balancing belt driven hydro-extractors of pivot type. The bags are placed on these 20 at a time, and the greater part of the liquid still adhering to them is wrung out. They are charged and discharged from the top by hand and the liquor is delivered from the outer casing into a dump, through a 2½ inch pipe connected to the base of the casing of the machine.

Driers.—There are two Ibis independent continuous drying machines of travelling band type. The whole machine is about 20 feet long and 10 feet wide, the walls and roof of deal lined on the outside with sheet zinc. In each of these the bags are clipped bottom upwards on to series of wooden hanger bars. These bars are screwed on to three endless chains which run on sprocket wheels fitted to steel shafts, one at each end of the drier, the whole having the effect of an endless belt or brattice

Every fourth bar has a wheel at each end which runs along, both going and returning on angle irons. The shaft at the feed end is fitted with a hand-wheel placed at one side of the drier. When a bar has passed right through the drier and begins to move upwards to return to the front, the clips fall by their own weight and release the bags, which fall opposite a glass sliding door in the back of the drier, through which they can be removed.

Air is blown into the drier by a belt-driven fan placed at the bottom of and at one side of the drier, and thence through a steam radiator placed near the back wall of the drier, which is completely covered by iron sheeting having a rectangular opening at the bottom perpendicular to the length of the drier, through which the heated air passes. As it emerges it passes through another uncovered radiator and travels through to the feed end in a direction counter to that of the bags. Two air-circulating fans are arranged above the endless chain of hanger bars, along the length of the drier, operated by a belt driven from the main fan shaft. Water condensed in the radiators and steam pipes, is blown up from steam traps to a reservoir placed on a platform elevated above the floor, from where the hot water can be fed to the various parts of the room as required, or overflow to the drain. The centrifugal extractors and drying machines are driven from a common line shaft by a 12 h.p. motor running at 720 r.p.m. and taking 17 amps. at 440 volts.

Eggs, filter process and reservoirs.- There are two cast-iron pressure eggs. Liquor from the boiling tanks is run through funnels fitted with a coarse metal mesh into a pipe feeding these eggs, and is blown from them by compressed air through either of two filter presses up to either of two steel reservoirs placed on the same platform as the tank for water condensed in the driers. The two cylindrical eggs of $1\frac{1}{4}$ inch cast iron are of the following internal dimensions, length, 3 feet $2\frac{1}{2}$ inches; diameter, 1 foot $2\frac{1}{2}$ inches. The two ends of the eggs are segments of spheres, the depth of each segment being $2\frac{1}{2}$ inches. The capacity is about 3·8 cubic feet. They are situated on the ground floor below and in front of the boiling tanks, and are filled by a 2 inch pipe which runs along the front of the tanks and has funnel-shaped openings fitted with coarse metal grids under the exits from the tanks. This pipe is fitted with two plug cocks, one governing each egg. A $1\frac{1}{2}$ inch pipe, also fitted with plug cocks, leaves the eggs at the bottom. Compressed air is introduced through $\frac{3}{4}$ inch pipes with air valves, and each egg is fitted with a $\frac{1}{2}$ inch exhaust pipe with valve.

The filter presses, through either of which the solution may be delivered from the eggs to the evaporator feed tank, are of the hand-operated type, consisting each of nine " pyramid " cast-iron plates of surface area 2·75 square feet. Flannel is placed between each pair of plates to form the filter proper. The liquid enters through a central hole and leaves by a narrow central outlet at the foot of the plates.

In order to prevent crystallisation in the pipes, it was found necessary to lag thoroughly the eggs themselves and the pipes leading from them

with steam-pipe coverings. For the same reason the filter presses are covered with detachable mats of non-conducting material, and the reservoirs are also lagged.

There is a liquor reservoir of the dimensions 3 feet 6 inches × 4 feet × 3 feet 6 inches, with a capacity of 49 cubic feet, which serves as evaporation feed tank. It stands on a platform above the evaporators, to which it is connected by a pipe leading from the bottom with two branches leading to the two evaporators. There is a main valve in this pipe, and a valve in each of the branches by which the flow to the evaporators is regulated.

There is another valve in each of the branches just at the entry to the evaporators. During normal running the main valve and two lower valves are turned full on, the upper valves being regulated to the desired flow. Between the main valve and the point where the pipe branches, there enters a pipe from the hot-water reservoir, fitted with a valve. By shutting off the main valve, either or both of the evaporators can thus be flushed out with hot water.

The liquor in this tank is made up of liquor blown from the boiling tanks and of mother-liquors brought up from the crystallising pans by a steam lift.

In addition to this reservoir there is a large tank (60 cubic feet capacity) not in the original design. This was installed for holding weak liquor from the boiling tanks from which it can be blown up after a charge of bags has been boiled, and to which it can be run back after the removal of the bags, this being repeated until the liquor is sufficiently concentrated. It can also act as an overflow reservoir to the evaporation feed tank, matters being so arranged that it is absolutely interchangeable with this tank, i.e. the evaporators can be fed and liquor can be run to the boiling vessel from either tank.

Evaporators.--Two horizontal Kestner emulseur evaporators are arranged under the reservoir platform. Each consists of two concentric steam pipes about 80 feet long, arranged in a space of about 20 feet long by the piping being folded over on itself three times. The piping is covered for the greater part of its length.

The liquor comes down from the reservoir by a pipe with two branches, one to each evaporator, entering at the bottom, and passes up through the apparatus. The steam enters at the top and passes along in a direction counter to that of the liquor. The liquor exit from each evaporator passes up vertically through the platform to a circular separator. The emulsion of steam and concentrated solution goes up through this pipe, into the separator, where the steam escapes by a pipe through the roof and the solution falls down through a pipe leading from the bottom of the separator to an iron launder, which carries the solution along to any of the three crystallising pans. Steam condensed in the apparatus is blown from a trap to a small tank on the floor.

Crystallising pans.--The crystallising pans consist of three rectangular jacketed iron pans, arranged side by side, each having three vertical sides, the fourth side, far from the liquor entrance, sloping up to a low angle. Water is run into the jacket at this end on each side of the pan,

and runs to the drain through a pipe from the bottom at the other end. The pans are arranged with a very slight slope towards the end nearest the launder, and in one corner is placed a draining hole close by a plug with an iron handle, through which the mother-liquor from the crystals can be discharged into an iron pipe running along the floor under the three pans. A steam ejector is fitted to one end of this pipe by means of which the mother-liquor can be lifted up to the reservoir again. The other end of the pipe can be screwed off to allow any mother-liquor to run to the drain if desired.

Each of the three pans is of the following internal dimensions :—

Depth - - - - - $9\frac{1}{4}$ inches.
Breadth - - - - - 4 feet 9 inches.
Length to where bottom begins to slope up 11 feet.
'Additional length - - - 3 feet.

The total capacity when brim-full is about 45 cubic feet.

The liquor coming along the launder is directed into the desired pan by blocking off the exits to the others with wooden gates made to fit closely into the launder.

A draining platform consisting of wooden bars $\frac{1}{4}$ inch apart and with a 3 inch catch-board all around, is fitted on to the shallow end of each pan.

Dry bag store.—This is situated near that end of the washing plant where the driers are placed. Dry bags are taken there on trolley rails leading from the back of the driers. They are then sorted into three classes, sound, split or ragged bags, which are then baled and stored. Fibre is put with the ragged bags. The capacity of the store is about 11,800 bags.

Operation of plant.—As previously stated, experience showed alteration in plant and working to be advisable. These alterations will be described later. The following notes refer to the operation of the plant as originally designed.

The procedure in boiling is as follows :—

' Liquor from trough 1 is levelled over and fills the boiling tank to a depth of about 18 inches. The inlet cock is then closed, the charge of bags (usually 50)* is introduced, and the necessary additional quantity of liquor from trough 1 is pumped in by means of a hand-operated rotary pump with a flexible delivery pipe. The pump is fed from the bottom of trough 1. The bags are not folded, but put into the boiler haphazard as it was found that if the bags are folded a large amount of nitre is left between the folds at the end of the boil.

The bags are compressed only on introduction to the boiling tank, the piston being raised during the boiling. In from 10 to 15 minutes the liquor is in active ebullition at a temperature of from $105°-110°$ C., and this is continued for a further 30 minutes.† At the end of this

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* The charge was originally 80 bags, but it was found that the tight packing necessary to immerse them prevented a proper circulation of the liquid and an even distribution of temperature.

† Later, the time of boiling was gradually increased to 1 hour, as shown on the flow-sheet, Fig. 32.

time steam is turned off and the liquor is run to the eggs and blown up to the new liquor tank, the piston being down to express as much liquid as possible from the bags. The bags are then removed from the tank and placed in trough 1 (special sticks fitted with blunt, twisted hooks (shunter's hooks), are used for this purpose), where they are allowed to soak until the trough is full, a period of about $1\frac{3}{4}$ hours.

A new charge of bags is then placed in the boiling tank, and the liquor in the liquor tank is allowed to run back into the boiling tank. Boiling is started and the process is repeated until the liquor is sufficiently concentrated (five lots of 50 bags each generally suffice), when it is blown to the other liquor tank for feeding the evaporators, and a fresh lot of liquor from trough 1 is introduced into the tanks. A convenient strength of solution is one of sp. gr. $1\cdot37$ to $1\cdot40$ at $80°$ C.

From a number of experiments, the steam consumption in boiling was estimated as $1\cdot16$ lb. per bag. The estimation was based on measurement of the orifice in the valve through which the steam escapes to enter the boiler, and the time the valve is open during the boiling. The amount of steam passing through the valve open to different extents was found by passing the steam into cold water and measuring the amount condensed. The results thus obtained were checked by passing steam into known quantities of water and noting the rise in temperature.

Soaking.—Bags are left in each trough for about $1\frac{3}{4}$ hours, so that they take about 7 hours to pass through the system. Leaving trough 4 they are placed on a large draining board until no more liquor drains away from them, before being thrown on the floor in front of the centrifugals.

Wringing.—The bags are put into the centrifugals in lots of 20, packed round the sides as evenly as possible. The amount of liquor removed per bag on the centrifugals is about 3 lb. As this liquor contains 12 per cent. of nitre it is collected and returned to the system, being pumped back into trough 3.

Drying.—The time taken in drying is about $3\frac{3}{4}$ hours.

The average temperature of the interior of the drier is about $50°$ C. Air leaves the radiators at a temperature of about $60°$ C. and issues from the machine at a temperature of about $39°$ C.

The consumption of steam in the driers was estimated by measuring the water condensed and blown up from the traps to the reservoirs. A number of determinations show an average consumption of steam of 406 lb. per hour. A correction for the amount of steam condensed in the pipes leading to the driers of $13\cdot13$ lb. per hour leaves the actual steam used in the driers as $392\cdot9$ lb. per hour.

It was found advisable to close down the radiator casings considerably with pieces of sheet metal, in order to limit the amount of air passing through the driers, as considerable heat was being lost through more than the necessary quantity of air being blown through.

Evaporating.—The liquor in the evaporation feed tank is made up of liquor blown from the boiling tanks and of mother liquors brought up from the crystallising pans. The specific gravity of this liquor is about 1·4, and it contains 46 per cent. solid in solution. Its average temperature is about 40° C.

The average temperature of the emulsion of steam and solution leaving the evaporator is about 110° C. The two evaporators together, running continuously, handle about 10 cubic feet of liquor in the feed tank per hour, liquor containing 46 per cent. of solid matter in solution, and of specific gravity about 1·4 at 40° C. They deliver liquor containing about 56 per cent. of solid in solution to the launder, at a temperature of about 85° C. to 90° C.

Weight of liquor charged per hour $10 \times 62 \cdot 26 \times 1 \cdot 4$ lb.

$871 \cdot 6$ lb.

Weight of solution discharged per hour $= \dfrac{871 \cdot 6 \times 46 \text{ lb.}}{56}$

$= 716$ lb.

Weight of water evaporated per hour $= 155$ lb.

Scaling of the evaporators is a source of trouble. To lessen this, the use of water with scale forming properties for washing should be avoided. It can be arranged that both drier traps and both evaporator traps deliver into a condensed water tank, from which hot distilled water can be fed to the system. Even when distilled water is used for washing, however, considerable scale still forms in the pipes and it is necessary to clear them out frequently.

Brass valves were originally used on the feed pipes to the evaporators, but the brass valves were later replaced by cast-iron ones as considerable trouble was being caused by the excessive corrosion of the feed pipes, and it was suspected that galvanic action was taking place.

A steam pressure of 45 lb. is usually maintained in the outer tubes, but slight variations are made as necessary to secure the requisite concentration.

The steam consumption has been estimated by collecting and measuring the water condensed in the apparatus. A number of determinations give the average weight condensed per hour as 212 lb.

A correction must be introduced for the water condensed in the steam pipes, and for the radiation from the evaporators themselves. The former is 4·7 lb. per hour. Owing to the difficulty of not being able to measure the temperature on the inside of the steam pipe constituting the outer layer of the evaporator, the correction for radiation has been made by assuming that the evaporator behaves, as regards condensation, like an ordinary 3 inch steam pipe. Each evaporator consists of 74 feet 6 inches of 3¾ inch pipe, of which 65 feet is covered with 1½ inch packing. The steam condensed per foot length per hour of a 3 inch pipe with 1½ inches covering is 0·11 lb. (*see* note on steam consumption later).

Steam condensed for an uncovered 3 inch pipe per foot length per hour is 0·79 lb.

∴ Total water condensed in evaporators due to radiation

$= 65 \times 2 \times 0\cdot11 + 95 \times 2 \times 0\cdot79 = 29\cdot3$ lb. per hour.

∴ Steam used in actual heating of the liquor

$= 212 - 4\cdot7 - 29\cdot3 = 178$ lb. per hour.

Crystallising.—Each pan is allowed to stand until the liquor has cooled to about 20° C. The liquor is then allowed to run out into the iron pipe beneath and taken up to the top reservoir by means of the steam ejector.

The crystallised nitrate is " fished " to the shallow end of the pan on to the draining rack, where, after a few hours draining, the moisture is reduced to about 6 per cent., in which condition the nitre is "bagged" and delivered to the Cummer drier attached to the retort house. The crystals vary in appearance, but in the majority of cases are of a marked bluish colour in bulk. Analysis, however, shows a purity of about 98·99 per cent. after eliminating moisture.

Steam consumption.—Steam is introduced into the plant by a main pipe running through the raw bag store and down to the centre of the main building. Before the pipe enters the bag store a pipe takes off any water condensed up to that point.

Another trap situated at the other end of the main pipe removes a portion of the water condensed in the pipe, but not all of it.

From this main pipe leads are taken off to the boiling tanks, the evaporators, and to each of the driers. In estimating the steam consumption for the various parts of the plant, an allowance is made for the condensation in the main and in the various leads. The improved practice described later would involve a smaller steam consumption, but below is shown the method by which an attempt was made to arrive at a figure for the steam consumption per bag.

The steam main consists of 66 ft. of covered and 3 feet of uncovered 3 inch pipe, and 33 feet of covered and 3·5 feet of uncovered 2 inch pipe. The condensation per foot length per hour in a 3 inch pipe with a 1½ inch cover is 0·11 lb., and for the same pipe uncovered is 0·79 lb. For a 2 inch pipe with a 1½ inch cover it is 0·68 lb., and uncovered 0·56 lb.

The total condensation in the mains is thus : (66 × 0·11) + (3 × 0·79) + (33 × 0·08) + (3·5 × 0·56) lb. per hour, *i.e.* 14·23 lb. per hour.

The amount delivered by the trap at the end of this pipe (found by measurement) is 6·0 lb. per hour.

∴ the amount of water condensed in the steam mains introduced to the various parts of the plant is 14·23 — 6 = 8·23 lb. per hour.

The two driers are fed by 42 feet of 1¼ inch covered pipe and 15 feet of 1¼ inch uncovered pipe.

The condensation per foot length per hour for $1\frac{1}{4}$ inch pipe with $1\frac{1}{2}$ inch cover is $0\cdot065$ lb., and for the same pipe uncovered is $0\cdot40$ lb.

$\therefore$ Condensation in leads to driers $= (42 \times 0\cdot065) + (15 \times 0\cdot40)$ lb. per hour

$$= 8\cdot73 \text{ lb. per hour.}$$

The evaporators are fed by 16 feet of 2 inch covered pipe and 2 feet of 2 inch uncovered pipe.

$\therefore$ Condensation in evaporator leads $= 16 \times 0\cdot08 + 2 \times 0\cdot56$ lb. per hour

$$= 2\cdot40 \text{ lb. per hour.}$$

Now, the total condensed water delivered from drier traps $= 406$ lb. per hour, and the total condensed water delivered from evaporator traps is 212 lb. per hour.

Total steam used per hour in boiling tanks (calculated from assumption that 200 bags are treated per hour at a consumption of $1\cdot16$ lb. per bag), 232 lb. per hour.

Of the $8\cdot23$ lb. water condensed in the steam mains and introduced to the various parts of the plant, it is assumed that quantities proportional to the steam consumption of the various parts of the plant are introduced to these various parts, i.e. of this $8\cdot73$ lb. the quantity received by the

$$\text{driers} = \frac{8\cdot73 \times 406}{850} \text{ lb. per hour}$$

$$= 4\cdot4 \text{ lb. per hour.}$$

$$\text{The quantity received by the evaporators} = \frac{8\cdot73 \times 212}{850} \text{ lb. per hour.}$$

$$= 2\cdot3 \text{ lb. per hour.}$$

The total amount of steam condensed in the main and leads introduced into the driers is thus $(4\cdot4 + 8\cdot73)$ lb. per hour, or $13\cdot13$ lb. per hour, and in the case of the evaporators it is $(2\cdot3 + 2\cdot4)$ lb. per hour, or $4\cdot7$ lb. per hour.

These amounts are deducted from the total amounts condensed in these two pieces of apparatus, as shown by measurement.

In the case of the boiling tanks no allowance is made for steam condensed in the main and leads, as in this case the estimation of the steam used was not based on the measurement of condensed water.

From the figures given in the various earlier parts of this report, the net steam consumption is—

In the boiling tank	-	-	$1\cdot16$ lb. per bag
In the driers	-	-	$392\cdot9$ lb. per hour.
In the evaporators	-	-	$178\cdot0$ lb. per hour.

Taking the number of bags treated as 4,000 per day, then—

Steam consumption in the driers $= 2\cdot36$ lb. per bag
Steam consumption in the evaporators $= 1\cdot07$ lb. per bag
Total net steam consumption on the plant $= 4\cdot59$ lb. per bag

Quantities dealt with.—The amount handled has varied a good deal. With the earlier working the average was about 4,000 bags per day of three shifts, but this number was reduced later, as it was found advisable to increase the time of cycle.

The weights of the raw bags as they come in, and also of the cleaned and dried bags, vary considerably, but the average figures given below may be taken as being not very far from the truth. The average moisture per raw bag has been determined in a large number of cases and on various days, wet and dry, by putting raw bags through the drying machines. The amount of jute fibre and earthy matter which is lost in transit has been determined by weighing all collected on the plant over a period of two days, and noting the total number of bags passing through the plant in that time.

	Lb.
Average weight of unbrushed bag - - -	4·50
Average weight of nitre recovered per bag by brushing -	1·20
Average weight of brushed bag entering the boiling tank	3·30
Average weight of moisture in bag entering boiling tank -	0·70
Average weight of fibrous or earthy matter deposited per bag in the plant - - - - - -	0·01
Average weight of washed and dried bag and NaNO$_3$ -	1·50
Amount of removable nitre per brushed bag - -	1·00
Amount of nitre recovered per brushed bag by plant -	0·85
Loss of nitre per bag - - - - -	0·24

The average amount of nitre in the washed and dried bags is about 6·9 per cent. or 0·104 lb. per bag. Variations in the treatment of the bags did not diminish this, and since the residues of nitre left appeared to be constant no matter whether fresh nitre bags or dump bags (which, owing to exposure to the weather, had a lower nitre content) were treated, the suggestion was put forward that nitre was present in the bags in two forms, one loosely adhering, varying in quantity and comparatively easily removed; the other fixed in some way, proportional in quantity to the amount of fibre present and not easy of removal.

Labour.—The plant is worked with three shifts during the 24 hours, the number of girls per shift being 16.

The work is divided as follows :

BAG WASHING PLANT

Monthly Report—4 weeks ending 6 a.m. 20th March 1918.

Boilers working	-	-	3	Hours working	-	449·25
Evaporators working	-	-	2	„ „	-	573·5
Driers working	-	-	2	„ „	-	405·75
Centrifugals working	-	-	2	„ „	-	302·75

Charged.		Produced.	
Unwashed	20,965 bags	Dry bags -	28,144
For rinsing	3,550 „	String and fibre	0·6538 ton
357·8 lb. string :		Sodium nitrate	29·6830 tons
equivalent to -	381		

Stocks.

Unwashed bag store.		Dry bag store.			Sodium nitrate.	
			Bags.	String & fibre.		Tons.
In stock - -	3,192	In stock - -	5,688	1·8076	Stock -	nil
Received from TNT	—	Received from plant	28,144	0·6538	Produced	29·6830
Received from G.C.	33,868					
		Total -	33,832	2·4612	Total	29·6830
Total -	37,060					
		Issued to traffic -	—	-		
Issued to plant -	26,965	Other issues -	12,675	—	Issued	26·8125
Stock - -	10,095	Stock - -	21,157	2.4612	Stock -	2·8705
Total -	37,060	Total -	33,832	2·4612	Total	29·6830

Physical data.

Strength of liquor in store tank -   -   45·4 per cent. $NaNO_3$

Steam pressure in evaporators   -   48·3 lb.

Temperature of liquor leaving evaporators -   84·1° C.

Temperature of driers, No. 1 -   -   32·8° C.

Temperature of driers, No. 2 -   -   36·3° C.

Moisture in sodium nitrate produced   -   8·24 per cent.

Sodium nitrate in dry bags produced   -   6·6 per cent.

Sodium nitrate recovered by brushing   -   1·3719 lb. per bag.

Dry sodium nitrate recovered by plant   -   0·9695 lb. per bag.

Efficiency of plant   -   -   -   -   91·8 per cent.

1 charge hand, who is in charge of the shift, and who works the eggs and evaporators.

1 girl bringing in bags to the boilers.

2 girls in charge of boiling tanks.

o girls unloading from the boiling tanks and passing bags through the four troughs

2 girls in charge of centrifugals.

2 girls charging driers.

2 girls unloading driers.

Other duties, such as removing scum from the surface of the liquor in the feed tank, cleaning and swilling down the floor and placing nitre from the pans on to the draining boards and into bags, are carried out as required by girls taken from the troughs.

The plant is in charge of a shift officer, who is on duty during the day from 9 a.m. to 5 p.m.

On page 75 a bag washing plant report, and in Fig. 32 a flow-sheet showing physical data, are reproduced.

Improved practice in bag washing.—Plant difficulties at one factory, together with an increasing stock of bags to be washed, made necessary

a deviation from the standard method, and these brief notes indicate the method adopted and conclusions arrived at with the results for six months from January 1st to June 29th, 1918.

Summary of results.—1. Nitre bags are thoroughly brushed at the nitre shed before delivery to the plant. Over long periods of averages the nitre content is estimated at 1·1 lb. per bag, whilst the average content for the six months is taken as 1·2 lb. per bag.

2. Boiling tanks have been removed as being unnecessary for the purpose of effecting the solution of the crystallised nitre on the bag.

3. Solution of nitre is effected by soaking the bags for 5–10 minutes in washing trough No. 1 at a temperature of 70°–80° C., demonstrating that prolonged boiling is unnecessary. Heat is supplied by the separated steam from the evaporators which had been allowed previously to pass out through the roof.

4. Full advantage is taken of the sponge-like properties of wet jute in the dilution and extraction of nitre.

5. It was found that it is possible on large operations to wash bags so that the percentage of nitre retained is less than 1 per cent, which would tend to disprove any theory of adsorption, which has been put forward as the reason of the high nitre content of washed bags.

6. Eggs and filter presses have been abandoned as unnecessary since the clarification of the solution is effected by settling.

7. Evaporation troubles have been classified under two causes—

(*a*) Mechanical,
(*b*) Chemical,

and to a great extent eliminated so that no appreciable scaling is now in evidence.

8. All hot condensed water from the driers is returned to the boiling tanks, so that by preheating the feed water, one Cochran boiler has been able to run the whole plant at its present rate of output.

9. One of the presses has been installed as an improvised baling machine, and all bags are now graded and wired in bales of 50, so that considerable saving of space in storage and transport has been effected.

10. 199,075 bags have been washed, dried, and stored during the period, which represent an average over the whole period of 1,103 per shift, or 138 bags per hour. The nitre recovered over the period was 86 tons, equivalent to 0·97 lb. per bag showing a loss of 0·23 lb. per bag on the estimated content of 1·2 lb., *i.e.* a loss of 19·1 per cent.

11. A flow diagram of the present process (Fig. 33), together with outlines of a model process and a comparative chart of the nitre content of bags at various stages, are reproduced herewith.

It appears—

(*a*) That a simple process has evolved which the average forewoman of ordinary intelligence can manage with a minimum of supervision.

(b) That it would be possible to construct a plant capable of treating at *least double the number of bags per shift* with less service and maintenance charges than in the standard type of process previously described.

(c) That with very little alteration and expense all existing standard plants could be converted as shown in Fig. 33, Process No. 2.

IMPROVED BAG-WASHING PLANT

Outlines of a model bag washing plant.—The bags are well brushed before being delivered to the plant and now contain an average of $1 \cdot 1$ lb. of nitre mostly in the crystalline state.

The first point studied was that of the method of effecting the solution of the crystallised nitre on the bag.

On the authority of Landolt Bornstein, the dissolving of one gram-molecule of $NaNO_3$ in 200 gram-molecules of water, absorbs 5 kilo-calories of heat; in the absence of data at greater concentrations this figure is taken as a basis of calculations, viz., 200×18 grams of water dissolving 85 grams of $NaNO_3$ absorb 5 kilo-calories.

One gram of water as steam condensing liberates 550 calories; therefore, for every 85 grams of $NaNO_3$ dissolved, 9 grams of water as steam are needed.

For every 85 grams of $NaNO_3$ recovered from a 50 per cent. solution, 85 grams of water must be evaporated as steam, so that there is available the heat of condensation of (85–9) grams of water as steam for general heating purposes over and above that required for the solution of the nitre.

According to the standard practice, 50 bags irregularly packed are agitated for 45 minutes at a temperature of 105°–110° C. using live steam for agitation, and a liquor of $1 \cdot 37$ gravity is obtained which is run to the evaporators.

Taking a solution of this strength as a basis, it was found that if bags were dipped loosely with slight agitation at a temperature of 80°–85° C., the whole of the nitre was in solution after five minutes, and any prolonged boiling was unnecessary.

Under plant conditions the bags are allowed 10 minutes soaking, which is ample for the solution of the nitre. This is demonstrated in the laboratory experiments mentioned later.

The plant consists of two units of wrought-iron trough, 18 feet long $\times$ 3 feet wide at top $\times$ 2 feet 10 inches high, with a capacity of 90 cubic feet, heated by passing the separated steam from the evaporators through a grid of 3 inch wrought-iron pipes fastened to one side of the trough so as to promote slight circulation of the liquors, any uncondensed steam being allowed to escape through the roof by means of a small stack.

In this manner a temperature of 70°–80° C. is maintained in these troughs whilst evaporation is in progress, and 1 inch live steam connections are let into the inflow pipes to provide heating if at any time no evaporation is taking place.

Forty bags are loosely dipped in the solution in No. 1 trough and allowed to soak for 10 minutes, and are then transferred to draining board No. 1. This is repeated until the solution reaches a specific gravity of 1·30 at 70° C.

As the medium used for the recovery of the nitre from the bags is water, experiments were conducted to find the best method for eliminating this water from the bag after the solution of the nitre had been accomplished. The following table shows averages over a large number of bags treated with water in every case :—

How treated.	Average weight in lb.	Average water content in lb.
Wet and undrained - -	7·0	4·6
Wet and drained - -	4·5	3·1
Wet and mangled (hand) -	3·5	2·1
Wet and centrifuged - -	2·2	0·8
Dry - - - - -	1·4	—

This shows a margin of about 100 per cent. in favour of the centrifugal machine over mangling, allowing for a slightly larger extraction with power mangles. This means that a bag having an average nitre content of 1·2 lb., after boiling in 45 per cent. nitrate solution (as is the standard practice), would contain under similar conditions the following amount of nitre :—

How treated.	Approximate moisture content in lb.	Per cent. remaining.	Approximate nitre content in solution in lb.
Unwashed bags -	sp. gr.	100	1·2
Wet and undrained -	1·35 × 4·6 = 6·2	230	2·8
Wet and drained -	1·35 × 3·1 = 4·2	160	1·9
Wet and mangled -	1·35 × 2·1 = 2·8	105	1·36
Wet and centrifuged -	1·35 × 0·8 = 1·0	37	0·45

This figure depends on the size and speed of the centrifugal used and time factor.

Furthermore, a moist bag from the centrifugal machine when plunged into water immediately absorbs, with a sponge-like action, water up to 4·6 lb.; this in turn dilutes the strong nitre solution in the bag four to five times, and by again centrifugalling reduces the percentage of nitre in the bag in the same ratio.

One hundred grams of dry bag from the nitrate shed were heated for 10 minutes at 85° C. in 300 c.c. of water and then centrifuged. The fibre was again heated for 10 minutes at 85° C. in 300 c.c. of water and again centrifuged. This was repeated a third time, after which the bag was dried, weighed, and the nitrate content estimated.

Laboratory experiments on fresh bags

	Nitre extracted.	Nitre remaining.
	Per cent.	Per cent.
1st boil	50·2	·7·34
2nd boil	6·3	1·04
3rd boil	0·7	0·34

Another 100 grams of dried bag were heated for 10 minutes at 85° C., then centrifuged, then washed on the centrifugal whilst standing with 100 c.c. of clean water, and again spun. This operation was repeated three times, the bag weighed, and the nitre content estimated.

	1st Method.		2nd Method.	
	Extracted.	Remaining.	Extracted.	Remaining.
	Per cent.	Per cent.	Per cent.	Per cent.
1st wash	50·2	·7·34	47·2	5·85
2nd wash	6·3	1·04	5·2	0·65
3rd wash	0·7	0·34	0·5	0·15
		On dry fibre.		On dry fibre
	43·5	0·78	47·2	0·31

Washing on a centrifugal has to be done when the machine is at rest or travelling at not more than half speed, otherwise the compression of the individual fibres is too great to allow of the penetration of the wash water. On a plant scale as shown in flow diagram of No. 2 process, it has been possible to wash bags so that the nitrate content is well below 1 per cent.

From these experiments the following washing process was put into operation, and during the necessary reconstruction changes, the output of the department was maintained as steady as possible with the following cycle :—

Forty bags are dipped into washing trough No. 1, A or B unit, allowed to stay there for 10 minutes and then placed on draining board A 1 or B 1 for 10 minutes, transferred to washing trough No. 2, where they remain for 10 minutes, then on to draining board No. 2 A or B, for 10 minutes. Twenty are treated in centrifugals A or B, being spun for seven minutes and washed when starting with approximately 1½ gallons wash solution from washing vat No. 3. This is to replace the solution carried forward by the bags; all solution from centrifugals returning direct to washing trough No. 2. The bags are loosely thrown into washing trough No. 3, where they remain fo. 10 minutes, and then placed on draining board No. 3, transferred to washing trough No. 4

for 10 minutes and then given 10 minutes on draining board No. 4, finally being spun in machines No. 3 or 4, the solution from which is returned to washing trough No. 3. Cycle of bags as follows :—

Soaking -   -   -   -   40 minutes.
Draining -   -   -   - 40 ,,
Centrifuging   -   -   - 20 ,,

Thus bags are ready for drying in 100 minutes. When washing v; No. 1, A or B, has reached a specific gravity of 1·39 at 70° C., it is allow to settle for one shift, and then pumped to Kestner feed supply tanks for evaporation. No. 1 trough is then filled from No. 2 and 3 from No. 4; condensed water from Kestner being run into No. .; as required, and made alkaline with soda ash.

Two of the driers are worked with restricted radiator openings; the capacity of these is obviously controlled by the moisture content of the bags as they come from the centrifugals. With an overall spin of about 10 minutes, the present machines are capable of reducing the water content of 40 bags to 0·56 lb. per bag; but if larger machines were installed this would be materially reduced. However, it would probably be cheaper to enlarge and strengthen the present driers if additional capacity were required, since the present type is rather light for this class of work. The maximum output so far demanded has been 200 bags per hour for the two machines, which has not overtaxed their capabilities.

The bags when dry are graded 1st, 2nd, and 3rd, and pressed into neat bales of 50 and wired. By this means a large economy of space is effected in storage and transport.

One of the presses from the dismantled boilers has been improvised as a baling machine close to the delivery end of the driers, and the grader sorts and bales as the bags are dried.

There are two independent four-tube Kestner evaporators installed designed to work at steam pressure of 15 lb. These have 5   of evaporating tubing of 40·8 square feet of evaporation surface, and the usual vapour separators.

There is a large volume of low-pressure vapour separated from the evaporated liquor, as it is previously pointed out, which has been allowed to waste in the standard design of plant, and is available for heating purposes.

However, care must be exercised so that no back pressure is created on the evaporators which would retard their capacity for evaporation.

The vapour has been utilised for the following purposes :—

(1) To supply the necessary heat for the solution of the nitre on the bags in the primary washing vats.

(2) To preheat the solution in the evaporator feed tank.

The vapour is led through a tubular condenser of ample dimensions which has large condensing surfaces, and is provided with efficien draining pipes and free atmospheric outlet for all excess of vapour.

As previously stated, scaling of the evaporators has caused considerable trouble at most factories, and may generally be assigned to two main causes, viz. :—

(a) Chemical,
(b) Mechanical.

Chemical. –There appear to be varying quantities of calcium sulphate, either from the leachings of the nitre collecting in the bags, or as weighting material in the fibre of the bags. This material deposits on any heating surface, and very quickly reduces the heating efficiency.

To eliminate this undesirable impurity, soda ash was added to the solution in excess of the quantity required to precipitate all the calcium salts (calculated as $CaSO_4$) in solution as $CaCO_3$. This appears to have minimised the trouble, although it has not completely eliminated the scaling; as on examination of the tubes after $5\frac{1}{2}$ months' work, the three top tubes in one set were found to have a $\frac{1}{4}$ inch scale, and the corresponding tubes in the other had $\frac{1}{8}$-inch of scale. However, this scale was probably the result of deficiency of soda ash due to variations in the calcium salt content of the bags, and the analytical method of control has been abandoned. From mechanical reasons soda-ash is now added to the weak solution in the secondary washing vats Nos. 3 and 4, which are kept distinctly alkaline, and the evaporators are washed out daily with strong ash solution. It is anticipated that in this manner the evaporators will keep entirely free of this calcium scale.

The calcium carbonate so formed settles with the dirt and may be filtered out; the sodium sulphate is deposited probably as a mixture of crystals with the sodium nitrate. The system sodium nitrate sodium sulphate sodium chloride has not been worked out exactly, but a few figures may be given. A cold saturated solution of crude nitre, crystallising at $16°$ C. may contain 43 per cent. of $NaNO_3$, 3·5 per cent. of Na_2SO_4, and 4·0 per cent. of $NaCl$. As the $NaCl$ increases, the Na_2SO_4 is eliminated, so that there is usually obtained a cold saturated solution at $16°$ C. containing 39 per cent. of $NaNO_3$, 1·5 per cent. of Na_2SO_4, and 8·4 per cent. of $NaCl$. The maximum (SO_4) calculated as Na_2SO_4 found so far in recovered nitrate is 1·5 per cent., but the average content is about 0·8 per cent., representing roughly the addition of 14 lb. of ash per 100 bags treated. In factories which object to adding ash to the solutions owing to the presence of sodium sulphate in the nitre, the washing out and soaking in ash solution of the evaporators would be sufficient provided the rate of flow was maintained through the evaporators by either of the methods mentioned later under mechanical running.

Mechanical. –The average temperature of feed liquor, after dilution with return mother-liquor from crystallising pans, is about $50°$ C., and the initial boiling point is $108°$ C., with a final boiling-point at a concentration of 1·5 specific gravity of $115°$ C. This represents, therefore, a total heating range of $(115° - 50°) = 65°$ C., and an evaporating range of $7°$ C.

The steam pressure used averages 45 lb., with a saturation temperature of 145° C., so that the margin of difference from out-going liquor is (145° – 115°) 30° C.

From these figures it is evident that too much has been asked of these small heating surfaces, and consequently the rate of flow for high saturations has had to be materially reduced. Using them thus, as independent units, has materially aided in the mechanical scaling by reason of the velocity of liquor.

If the standard method of operating were to be made efficient, at least one six-tube evaporator should be installed to allow of a good flow of liquor. There are, however, alternative methods of working the present evaporators to effect similar results, for example, as follows :—

The two sets of evaporators may be worked in series, concentrating from a specific gravity of 1·34 at 19° C. to a specific gravity of 1·45 at 90° C. in No. 1 set, and feeding the concentrated liquor from No. 1 into a finishing off in No. 2 to a specific gravity of 1·50 at 100° C., so that the rate of flow is at least 30 cubic feet per hour instead of 10 cubic feet per hour as in standard practice.

The method now in use is a double circulation method by which all the liquors are daily circulated through the evaporators until the whole of the concentrated liquor reaches a specific gravity of 1·48 at 90° C., and then finally run to the crystallising trays at specific gravity 1·50 at 100° C., running at the rate of about 30 cubic feet per hour. By this means it is possible to obtain a yield of 4 to 6 cwts. of nitre per tray, so that per 8 hour shift it would be possible to recover from 8 to 12 cwts. of the nitre, allowing from 1 to 2 hours for soaking trays in ash solution and washing out. It is found that with the present recovery of 10 to 15 cwts. per day it is possible to close down the evaporators for eight hours for soaking when only working one 8 hour shift on bag washing.

With the elimination of the boilers the further use of the blowing eggs was unnecessary, and these have been removed. The efficient filtering of such small quantities of liquor with the type of filter press installed was a problem requiring very close attention. However, as all suspended matter in the liquor settles in a period of from 4 to 8 hours, varying with the depth and density of solution, the clear liquid may be decanted off by means of a tap about 4 inches from the bottom and the settled mud periodically cleaned out, so that by alternately using the two primary solution troughs, first as solution tank, and then as settling tank, the need for filter presses has been eliminated, and it has been found necessary to clean out each primary vat only once per week. By this method, the solutions run to the evaporators are remarkably clear and no fouling of the solution takes place, consequently the colour of the crystal recovered is generally good, and it is only found necessary to dispose of the residual mother-liquors once in two or three months.

The flow diagram (Fig. 33) is drawn up on different lines from that on Fig. 32 for period March 12th to 24th, and shows the bag which is the subject of treatment, at the varying stages during the process of washing under standard practice, and improved practice —No. 1 and No. 2.

Process No. 1 is the one described above, and is the one which can most economically be worked with the present plant lay-out. It was intended to have a power mangle installed instead of a draining board between washing troughs Nos. 1 and 2, but this has not been possible, so that washing on the centrifugal machine before compression of the fibre takes place has been installed as a compensation. Of the two centrifugal machines used, the steam driven has been the most useful.

The result of a week's working of the improved process are given below :—

No. of bags washed   -   -   10,146
Weight of $NaNO_3$ recovered - 12,452 lb. dry.

Average weight recovered per bag $\dfrac{12452}{10146} = 1 \cdot 23$ lb. per bag.

Average weight of brushed bag $+ NaNO_3 + H_2O = 3 \cdot 43$ lb.
There is $25 \cdot 8$ per cent. H_2O in a brushed bag.
Weight of $NaNO_3$ in brushed bag charged

     $= 3 \cdot 43$ lb. $-25 \cdot 8$ per cent.—weight of dry bag.
     $= 3 \cdot 43$ lb. $-0 \cdot 88$ lb.—weight of dry bag.

Average weight of dry bag $= 1 \cdot 43$ lb.—(moisture + $NaNO_3$ content).
Moisture $= 10 \cdot 8$ per cent.
$NaNO_3$ content $= 1 \cdot 98$ per cent.

        $\overline{12 \cdot 78}$

Hence average weight of dry bag $= 1 \cdot 43$ lb. $- 12 \cdot 78$ per cent.
           $= 1 \cdot 25$ lb.

$\therefore$ $NaNO_3$ charged per brushed bag $= 3 \cdot 43 - 0 \cdot 88 - 1 \cdot 25$
           $= 1 \cdot 30$ lb.

Efficiency of plant $= \dfrac{\text{Amount of } NaNO_3 \text{ recovered} \times 100}{\text{Amount of soda charged}}$

        $= \dfrac{1 \cdot 23 \times 100}{1 \cdot 30}$ per cent.

        $= 94 \cdot 6$ per cent.

COST OF RUNNING NITRE BAG-WASHING PLANT

Standard Practice.

Below are given some actual costs for the five weeks ending 1st June 1918, together with the corresponding figures for the previous month.

		Previous month.
Washed bags produced - -	40,874	40,358
Nitre recovered :—		
By plant	17·35 tons, or 0·922 lb. per bag	13·07 tons, or 0·835 lb. per bag.
By brushing	22·57 tons, or 1·198 lb. per bag	25·26 tons, or 1·593 lb. per bag.
Total	39·92 tons, or 2·120 lb. per bag	38·33 tons, or 2·428 lb. per bag.

Cost per 1,000 bags.

	Cost of production.	Present period.	Previous period.
	£.	£	£
(a) Plant officers - - - - -	50·217	1·229	0·802
(b) Operative wages (29·2 operatives) -	237·383	5·808	6·435
(c) Overtime allowances (time and half) -	13·070	0·320	0·436
(d) Yard and traffic - - - -			0·016
Steam and steam power (8,925 tons of coal) -	192·937	4·720	3·713
Plant general expenses :—			
(a) Plant stores - - - -			
b) Clothing - - - -	1·936	0·033	0·084
c) Shift house and mess room - -			0·011
Maintenance of plant and process buildings -			
(a) Materials - - - -	3·725	0·091	0·745
(b) Wages - - - - -	71·513	1·750	3·249
(c) On cost (shops and salaries) -	20·4	0·500	0·4
Total - - - -	591·191	14·451	15·857

Hence the cost for the month of May is £0·01445 per bag, of 468d. per bag; and the cost for the month of April is £0·01586 per bag, or 3·806d. per bag.

Washed bags are divided into three categories, known respectively as sound, slit, and ragged; the proportion of these that come through are very approximately as follows : -

60 per cent. sound, 39 per cent. slit, 1 per cent. ragged.

Taking sound bags as being worth 4½d. each, slit bags as being worth ₃d. each, and ragged bags as being worth £0·25 per ton of 1,400 bags, the average value of a washed bag might be taken as :-

60 per cent. of 4·5d. 39 per cent. of 3d. 1 per cent.

$$9\cdot25 \times \frac{240d.}{1400}, \text{ i.e. } 2\cdot70d. \quad 1\cdot17d. \quad 0\cdot016d., \text{ or } 3\cdot886d.$$

At a time when the above were the values of sound, slit, and ragged washed bags, an unwashed bag could be sold for 2d. Hence the value of the bag may be taken as having been increased by 1·886d. by washing. The value of wet nitre in May 1918 was £22·917 per ton, or 2·45d. per lb.

This gives 5·194d. as the value of nitre recovered per bag in May, and 5·949d. as the value of nitre recovered per bag in April; i.e., the profit on the plant was :—

In May (5·194 + 1·886)d. — 3·468d., or 3·612d. per bag
In April (5·949 + 1·886)d. — 3·806d., or 4·029d. per bag

This, of course, makes no allowance for amortization.

Improved practice.

For the initial working of the improved practice during a period of six months when 199,075 bags were treated, and the recovery of nitre per bag was 0·97 lb., the working costs were as follows :—

	£	s.	d.	d.
Power and light	-	73	13	5	0·69 per bag.
Steam	-	-	626	19	9	0·76 ,,
Wages	-·	-	1,078	12	1	1·3 ,,
Maintenance -	-	140	9	6	0·17 ,,
		1,919	14	9		2·32 ,,,

This includes the wages of a forewoman and 16 women workers, with the full salary of a chemist. This last item would be unnecessary, however, when once the process was in running order; and the labour, too, would be reduced.

The nitre recovered per bag is that recovered on the bag-washing plant only, the bags being brushed in the nitre shed before delivery to the plant.

Cost of alterations to plant made in order that the practice might be modified are also included.

These figures are sufficient to show that a nitre bag-washing plant can be run at a considerable profit where there are a large number of bags to be treated.